A Summary of Current Program, 7/1/62

and Preliminary Report of Progress

for 7/1/60 to 6/30/62

WESTERN UTILIZATION RESEARCH AND

DEVELOPMENT DIVISION

of the

U.S. AGRICULTURAL RESEARCH SERVICE

UNITED STATES DEPARTMENT OF AGRICULTURE

This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

There is included under each problem area in the report a brief and very general statement on the nature of the research being conducted by the State Agricultural Experiment Stations and the professional manpower being devoted by the State stations to such research. Also included is a brief description of related work conducted by private organizations. No details on progress of State station or industry research are included except as such work is cooperative with U.S.D.A.

The summaries of progress on U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1960, and June 30, 1962. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Albany 10, California.

UNITED STATES DEPARTMENT OF AGRICULTURE
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INTRODUCTION

Utilization research on agricultural commodities deals with the discovery and development of new and improved products and the invention or perfection of processing technologies. The scientists, engineers, and technologists who carry on this research devote a substantial part of their concerted efforts to basic studies of physical and chemical properties of the commodities and products derived from them, in order to provide a firm base of exact knowledge for applied developments.

The present report summarizes the current research program of the Western Utilization Research and Development Division (one of four Utilization Divisions in the Agricultural Research Service), makes a report of progress toward the objectives of that program during Fiscal Years 1961 and 1962, describes a few of the more significant recent accomplishments of this work, and summarizes utilization research conducted and projected by private industry, non-profit research organizations, and the State Agricultural Experiment Stations as of the end of Fiscal Year 1961.

Research Area Covered by this Report

The farm commodities dealt with in this report are the cereal grains wheat, rice, and barley; alfalfa and other forage crops; wool and mohair; citrus, apples, and other fruits and tree nuts; potatoes and other vegetables and dry beans and peas; castor and certain other oilseeds; sugar beets; new and replacement crops; and poultry and eggs.

Pharmacological research for all four of the Utilization Research Divisions is conducted at the Albany laboratory of the Western Division, and is described in this report.

Distinct phases of research on certain commodities having broad agricultural significance are pursued in others of the Department's Utilization Research Divisions besides the Western Division: Research on industrial uses of wheat and on milling technology is carried on in the Northern Division; certain areas of research on deciduous fruits and on potatoes and other vegetables are handled by the Eastern Division; particular lines of research on rice, vegetables, and fruits are carried on in the Southern Division. Research on new and replacement crops is carried on in all four Utilization Divisions.

Aims of Research on These Commodities

The group of commodities discussed here provides the nation with more than half of its food, either directly (cereal grains, fruits and vegetables, poultry meat and eggs, and beet sugar) or indirectly through feeding of meat animals (forage crops, wheat, barley). The other commodities in the group supply us with our most important animal fibers (wool and mohair) and offer opportunities of development into numberless industrial products (castor and other oilseeds).

Thus the general aim of utilization research on both of the two broad categories of farm commodities is essentially the same--to broaden and extend the avenues of utilization of the commodities and thereby help to stabilize or increase the demand for them. The scientific procedures of research are broadly the same in both areas, whereas the technologies are in many respects different, especially as between the food materials and all the others. The fundamental justification for carrying on a publicly supported program of utilization research on food products follows a somewhat different line of reasoning than the justification for research to extend the utilization of non-food commodities.

The latter may be based on the avowed public policy of assisting a farm commodity threatened with loss of its markets as a result of the swift rise of non-agricultural synthetics, as in the case of wool; or on the possibility of developing demand for a presently minor crop to the point where it can be grown profitably on a very large scale and thereby remove some of the pressure of surplus from other crops--the growing of castor and certain other industrial oilseeds being an example of this.

Research on the processing of farm products for food, on the other hand, is justified primarily by its direct benefit to the entire population through improved nutrition and well-being, reduction of economic losses resulting from spoilage and waste, and increased opportunity to find profitable markets abroad. Indirectly, too, advances in technology through food processing research are capable of bringing about major and desirable shifts in the commodity supply and demand picture for the country as a whole, as for example in the economical conversion of abundant feed grains into broiler-type chickens, marketable in refrigerated or frozen form throughout the nation--and, indeed, even overseas.

Organization of the Division

Research and development along these diverse lines are carried on for the Western Division by a staff headquartered in the Western Regional Research Laboratory, Albany, California. A smaller

Department-owned laboratory is operated in Pasadena, California, and laboratory space and facilities in Prosser and Puyallup, Washington, are utilized through a cooperative arrangement with Washington State University, Institute of Agricultural Sciences.

The Albany research staff is organized into six commodity-oriented Laboratories (Cereals, Field Crops, Fruit, Poultry Products, Vegetables, and Wool and Mohair); two functional Laboratories (Pharmacology, and Engineering and Development); and a Pioneering Laboratory concerned with basic studies of plant enzymes. The staff at Pasadena is organized as the Subtropical Fruit Laboratory. The Western Regional Research Laboratory, at Albany, also houses the Division Director's staff, the staff required for Administrative Management of the Division, and that responsible for Plant Management--that is, operation of the buildings, facilities, and grounds.

Division scientists and engineers not only conduct or supervise research in their own experimental facilities, but also greatly extend the scope and influence of their work by planning and supervising developmental activities carried on by cooperating private firms, processor organizations, or industry groups, and by arranging for needed lines of research to be conducted by well-qualified scientists elsewhere under research contracts. In addition, certain grants of research funds are placed with investigators in foreign countries; the cost of these foreign research efforts on behalf of American agricultural interests is borne by Public Law 480 funds.

Examples of Recent Accomplishments of the Western Utilization Research and Development Division

New Bulgur Process to Aid Wheat Exports. A new, continuous process has been developed by the Agricultural Research Service for the conversion of wheat into bulgur (parboiled wheat). This process, which operates at atmospheric pressure, is economical in heat and labor requirements and employs conventional, readily-available equipment. A large midwestern grain company is constructing a bulgur plant based on this method, while other companies are converting existing equipment. Present plans of the Department call for the movement of over 300 million pounds of bulgur into school lunch and other market-building programs abroad this year.

Foam-Mat Dried Foods. High-quality instant food powders, produced by a new method developed by Agricultural Research Service engineers, promise to substantially expand exports for U.S.-produced fruits, vegetables, and other agricultural products. The method of manufacture, "foam-mat" drying, has now become a commercial reality, with installation of two plant-scale units. A growing number of industry pilot plants attests to the likelihood of its extensive use

in the future. The new drying method involves whipping liquid foods into a foam, incorporating suitable stabilizers when necessary; spreading the foam on a belt or perforated tray; drying in a stream of warm air; and finally rolling the dried foam into free-flowing granules which may then be compressed. Enough of the foam structure persists through compression so that instant rehydration is combined with the desired property of high bulk density.

Dehydrofreezing of Fruits and Vegetables Gains Broad Acceptance. Dehydrofreezing, a method of food preservation developed by Department scientists whereby foods are partially dehydrated and then frozen, is now in commercial use. Several million pounds of dehydrofrozen apples are being produced each year for use in commercial bakeries. Dehydrofrozen peas, carrots, and potatoes are being manufactured in rapidly increasing tonnages and are becoming important export items. Three million pounds of dehydrofrozen pimientos were produced last year for use in cheese products. A large food concern has just completed a market test of dehydrofrozen baby foods, including fruits, vegetables, soups, meat dinners, and puddings.

In the process, dehydration is conducted to remove at least half of the water present to avoid the irreversible quality damage that occurs during late stages of complete drying. The reduction in product weight and volume achieved by partial dehydration results in large savings in costs of freezing, packaging, handling, and shipping. Fresh flavor, texture, and color are retained by keeping the product frozen. Less drip on thawing and easier moisture control during remanufacture are among the advantages of dehydrofrozen over conventional frozen foods. The fresh-product quality, the convenience and the relatively low cost of reconstituted dehydrofrozen foods assure expanding acceptance of dehydrofreezing as a method of food preservation.

Major Cause of Egg Spoilage Eliminated. Basic studies of the nature of bacterial growth in egg white have revealed that trace amount of iron counteracts the protective action of the conalbumin, a protein component of egg white, thus permitting spoilage. Applied studies based on this finding show that iron in the water used to wash eggs can be high enough to markedly influence spoilage. With wash water containing 5 to 10 parts per million of iron, a level commonly found in well water, spoilage is two- to four-fold that obtained with water containing less than 0.5 parts per million of iron. This discovery is helping egg producers and processors reduce the serious shell egg spoilage that accounts for a loss of approximately $20 million annually. Major egg producers are starting to treat egg wash waters to lower iron content to 0.5 parts per million in order to eliminate excessive spoilage.

Nutrients in Dehydrated Forages Protected by Chemical Stabilizer. The major producers of dehydrated forages have announced that their total production will henceforth be treated with ethoxyquin--a stabilizing agent whose use for this purpose was developed by Department scientists. It is estimated that 80 to 90 percent of the dehydrated forage produced in the United States will be protected with this antioxidant, preventing severe losses of valuable provitamin A, vitamin E, and poultry and egg pigmenting factors (xanthophylls). Cooperative studies conducted at State Experiment Stations have shown that incorporation of ethoxyquin in mixed feeds also prevents certain diseases in poultry and lambs, as well as rancidification of fats in feeds. Development and commercial adoption of the ethoxyquin treatment is the result of a joint research effort involving Department, State Experiment Station, and industry scientists. Extensive toxicity testing over a 5-year period led to Food and Drug Administration approval for the use of the compound in dehydrated forages and mixed feeds for all types of animals. A new commercially available water emulsion of ethoxyquin has reduced cost of treatment by one-half--to less than 50 cents per ton of dehydrated forage or mixed feeds.

New Process for Making Wool Shrink- and Muss-Resistant to be Commercialized. Last year the Agricultural Research Service reported it had developed an entirely new method for making wool fabrics shrink- and muss-resistant, and that the method was being evaluated by a number of industrial firms. One of the large wool fabric manufacturers who treated several thousand yards of fabric by this method and evaluated it in a market survey, is purchasing more suitable processing equipment and will begin large-scale commercial production in early 1963. Because the treatment does not harshen or weaken the fibers or change the original texture of the fabric, as do some of the treatments now in use, potential applications cover a broad range of different kinds of woolen and worsted articles, and the number of manufacturers evaluating the treatment continues to grow. The name WURLANIZE has been chosen for this new process, WUR coming from Western Utilization Research and Development Division and LAN from lana, the Latin word for wool.

Area 1 WHEAT AND BARLEY-- FOOD AND FEED PRODUCTS AND PROCESSING

Problem. In recent years huge stockpiles of wheat and barley have accumulated, exerting a severe depressing effect on the agricultural economy through restricted grower incomes and expensive government control programs. The most promising solution to this critical problem lies in greatly expanded exports to meet the urgent food needs of large segments of the world's population and to secure an increased share of prevailing and future dollar markets for these grains. Knowledge and skills do not now exist to reach these goals. Ways must be found to adapt U.S. winter wheats and flours to the specific use requirements in Western Europe, currently representing a large potential dollar market. New food products from wheat must be created to fit specific needs and preferences of individual countries throughout the world. Simple inexpensive methods must be devised for use in developing countries to process U.S. wheats into products appropriate for their socio-economic structures. Greatly expanded scientific knowledge of the composition and processing properties of wheat and barley is essential to accomplish these important objectives. More complete knowledge of the chemical and physical properties of both the major and minor constituents of the grains, and of the changes that occur among them during processing, is needed to point the way to the new food and feed products and to new processing technologies. A thorough exploration must also be made of the inherent versatility of these grains as food and feed substances to achieve the utmost of their wide use potentialities.

USDA PROGRAM

A broad program of basic and applied research on wheat and barley is being conducted by the Western Utilization Research and Development Division at Albany, California; under contract at Pullman, Washington, Cambridge, Massachusetts, Lafayette, Indiana, Corvallis, Oregon, and Ames, Iowa; and under P.L. 480 research grants in England, France, Poland, Italy, and Israel.

Basic studies are concerned with characterizing the soluble proteins (albumins and globulins), gluten proteins, lipoproteins and lipids in wheat and flour, and identifying interactions in and between these substances; and characterizing the biologically-active compounds present in bran and germ. Different varieties and classes of wheat are being studied to determine intrinsic differences between the relatively scarce high quality bread-baking wheats and those surplus wheats which require chemical treatments to make possible their use for bread production. Applied research is being conducted on new

and improved food and feed products and processes, with emphasis on the development of products to help fill the food deficit in overseas countries.

The Federal program of research in this area totals 28.0 professional man-years.[1] Of this number 17.1 are assigned to chemical composition and physical properties; 10.0 to new and improved food products;[2] 0.6 to new and improved feeds; and 0.3 to new and improved feed processing technology. In addition, the Division sponsors 28.8 professional man-years of research under P.L. 480 including 22.8 on basic studies and 6.0 on applications of research findings.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in the North Central and Western areas in 1961, reported 6.6 professional man-years divided among subheadings as follows: Chemical composition and physical properties 5.2, new and improved foods and food and feed processing technology 1.4. Basic studies underway are concerned with the compositional, biochemical and physiological factors which influence technological properties of wheat. These studies should yield information on the basic differences between the hard, soft and durum wheats which are under investigation. Applied research involves investigation of the causes and effects of moisture translocation in canned baked goods, development of non-staling bread-like products, development of mixing specifications, and milling studies.

Industry and other organizations including milling companies, baking companies, grower organizations, and allied industries such as suppliers, conduct research programs that are predominately concerned with specific applications to individual corporate problems. The predominant scientific activity in industry organizations is concerned with quality control of products and raw materials, with a

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1/ In addition, a program equivalent to 7.0 professional man-years, including 3.1 for improvement of the wheat bulgur wafer for civil defense food stockpiles and development of food adjuncts to supplement the use of wheat bulgur wafers under conditions of emergency use and 3.9, by contract, for a study of factors affecting stability of wheat bulgur wafers was initiated with funds transferred from the Office of Civil Defense, Department of Defense.

2/ Including non-recurring funds equivalent to 3.9 professional man-years from the Administrator's F.Y. 1962 contingency appropriation, used to initiate a contract project on development of protein-rich, water-dispersible export wheat food products; and industry support by way of salaries provided by The Farmers Co-Operative Commission Company for two employees developing new wheat food products.

somewhat lesser emphasis on product and process development and improvement. Virtually all of the research information obtained is confidential or protected by patents. Published information most often concerns laboratory procedures or fundamental findings. All of the companies keep themselves well informed on the progress of research conducted in government laboratories, but their greatest demand is for fundamental information. Grower organizations who are expending modest sums to support research work on wheat, in contrast to commercial companies, seek more information on new and improved products from government laboratories. Estimated annual expenditures in this area by industry and other organizations are equivalent to 160 to 200 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Soluble Proteins and Sulfhydryl Groups in Flours. High-speed, and continuous dough-mixing equipment now in use by modern bakeries demands unusually high-grade wheat flours. Only 10 to 30% of our normal wheat crop is regarded by the industry as being suitable for use in this machinery. Why some flours will withstand severe mixing and others will not, is not known. Results of basic research on wheat proteins are beginning to offer some explanation for this difference.

In research conducted during the past year, the presence in flours of a lipoxidase enzyme capable of oxidizing sulfhydryl groups was demonstrated in model experiments using a low-grade (5th break) flour shaken in a slurry with added glutathione and safflower oil, the latter being the substrate for the enzyme. Heating and/or deproteinization of the flour greatly reduced the extent of oxidation of sulfhydryl groups (those in glutathione plus those in flour) during a fixed reaction period. Substitution of methyllinoleate for the safflower oil more than doubled the rate of oxidation of the sulfhydryl groups so that 82% of these had disappeared after two hours of shaking at room temperature.

Comparison of gel-electrophoresis patterns of water-soluble proteins from a Rio variety flour and a high-protein air-classified fraction from it, showed no qualitative and only minor quantitative differences. This contrasts with some qualitative differences observed among patterns from Rio, Brevor, Burt, and Omar (a club wheat) variety flours.

The results obtained in these studies, provide guidance to efforts to produce and provide flour blends of optimum quality for specific uses. They suggest that air-classification milling, which up to the present time has been applied largely to intermediate type wheats,

may have important applications in the Pacific Northwest. The currently reported work also provides means for obtaining valuable materials for basic research on correlation of chemical and physical composition of wheat with baking properties of flours derived from them. (Research in this field relating to new and improved food products is reported below.)

Contract studies underway at Washington State University are aimed at developing rapid and simplified methods for determining individual flour proteins. A variety of electrophoretic and paper chromatographic techniques are being investigated with respect to suitability for analytical separations of proteins in flour extracts for the purpose of quantitative estimation of individual components. Particularly promising results were obtained with disc electrophoresis in polyacrylamide gels. Densitometry of stained protein bands in gels appears entirely feasible. Present results indicate that variations in dye-binding capacity by different components can be determined by accessory use of the optical system of the Perkin-Elmer free boundary electrophoresis apparatus for determination of protein concentrates in unstained gels. Results can then be compared with values obtained by routine methods for stained proteins. Extraction of soluble proteins from flour is improved by prior removal of lipids with water-saturated butanol. Procedures for extraction and separation of albumin and globulin as groups require additional refinement to reduce cross-contamination.

2. Relation of Sulfhydryl to Enzymes in Wheat. Basic studies on the relation of sulfhydryl groups to the amylolytic and proteolytic enzymes in wheat, flour and malted wheat are being carried out under P.L. 480 at the University of Poznan, Poznan, Poland. Some of the accomplishments of particular value include observation of rapid loss of sulfhydryl from ground whole wheat; elucidation of precautions required to get reproducible results in sulfhydryl determinations; improvement in precision of proteolytic activity assay; and demonstration of a definite, but limited reduction of proteolytic activity in the presence of sulfhydryl blocking reagents.

3. Immunochemical Analysis of Wheat and Barley Proteins. Pioneering research in this unexplored field is being carried out under P.L. 480 at Pasteur Institute in Paris. The objective of these investigations is to develop sensitive and precise immunochemical methods for establishing correlations between the presence or amount of different wheat or barley proteins and derived products. Each protein preparation is injected into several rabbits, and the serum of these rabbits is then pooled and concentrated for electrophoretic studies. Excellent progress is being made in a difficult unexplored field.

4. Flour Lipoproteins. Research on lipoproteins is aimed on determining the fundamental chemical and physico-chemical basis for the

behavior of flour doughs and batters during mixing, fermentation, machining, and baking. The lipoproteins are important in various ways and evidence indicates they may play a dominant role in dough rheology.

The decreased solubility observed for gluten in acetic acid or aluminum lactate buffer which occurs after extraction of gluten lipids with wet butyl alcohol followed by acetone was found also to occur if the acetone wash is omitted. Variations in exposure time to the butyl alcohol and in the state of subdivision of the gluten particles, had little, if any, effect. The portion of gluten made insoluble in acetic acid can be dissolved in strong formic acid. Upon electrophoresis in aluminum lactate, the redissolved material appears to consist almost entirely of glutenin and the alpha components of gliadin.

Low- and high-protein fractions obtained by air-classification of a commercial straight grade flour from club wheat, had lower (1.4%) and higher (4.4%) total lipids, respectively, than the original flour (1.8%). On a unit protein basis, however, the percentage of lipids fell as the protein content increased. Both flour fractions had higher percentages of complex lipids and lower nitrogen to phosphorus ratios in their total lipids than the original flour. Somewhat more galactolipid plus lipoprotein was found in the complex lipid fraction of the low-protein flour fraction than in that of the high-protein fraction. Such information is useful in characterization of the wheat proteins as they occur naturally in the kernel. One form adheres tightly to starch granules and is concentrated in low protein fractions. The other occurs in interstices between granules and becomes concentrated in high protein fractions during air classification. Both forms appear to be intimately associated with lipids as they occur in the kernel.

In studies on hard red spring (HRS) wheat, it was found that lipids in a gluten washed from the flour amounted to about 9% dry weight of the gluten. Separation into classes yielded slightly more compound than simple lipids, with the phosphorus and most of the nitrogen in the compound lipids. These distributions are like those for the flour from which the gluten was prepared and the air-classified club wheat flours mentioned above. The nitrogen to phosphorus ratio was lower in the gluten than in the HRS flour lipids. Fractionation of compound lipids from the gluten and from the high- and low-protein air-classified flours, all gave 6 to 7 separated fractions based on phosphorus content. As the protein content of the original sample increased lipid sugars measured as galactose, decreased. As their polarity increased the fractions of each lipid generally contained more phosphorus and nitrogen. Sugar contents of the fractions increased to a maximum at the third to fourth fraction, then fell to

low values in the final fractions. Ester content showed an inverse trend with lowest values in fractions three and four. This trend was less evident with gluten than with flour lipids.

It was found that considerable non-lipid material (sugars, amino acids, peptides) remains in flour lipids even when dissolved in dry petroleum ether, and appears to interfere with fractionations. Washing chloroform-alcohol solutions of lipids by diffusion into aqueous 0.02% calcium chloride (Folch procedure) has been found useful to remove non-lipids.

The Folch-washed lipids of two hard red spring wheat and two hard red winter wheat flours were separated into neutral and polar lipids. Percentages of the latter were slightly higher in hard red winter (HRW) than in HRS flour lipids. Fractionation of the four polar lipid mixtures on silica gel provided very similar general distribution of components in each case, with minor but perhaps important differences. Analytical evaluation of these differences is in progress.

In order to identify lipoprotein components of gluten separated by starch-gel electrophoresis, glutens and gluten fractions in aluminum lactate-urea solution were pre-stained with Sudan Black B. Only the glutenin fraction, which did not migrate into the starch gel, was stained.

These findings suggest that appreciable differences in the ratio of glycolipids to phospholipids occur among flour and gluten lipids depending upon the protein content of the original sample. Such differences in composition could be expected to affect baking or other performance characteristics.

5. Whole Wheat Lipids. Basic studies on the composition of the lipids of whole wheat have very recently been undertaken under P.L. 480 at the Ecole Nationale des Industries Agricoles et Alimentaires in Paris, France. These studies seek more precise and complete knowledge of these lipids and their reaction to processing treatments.

6. Wheat Endosperm Constituents. Fundamental research on the constituents of wheat endosperm is designed to show the location and activity of various protein and cell wall components in the endosperm. Inevitably this research will lead to a better understanding of the perplexing differences in physical properties of flours, doughs and batters from different wheats.

Soluble proteins were extracted from air-classified fractions of Rio (hard red winter) wheat flour. Dialyzed extracts were chromatographed using gradient elution. In all runs, there was a major fraction of

essentially unadsorbed material. Runs at higher pH's showed two major and two to three minor peaks from the gradient elution with no important differences between the flour as-milled, coarse residue, fine and intermediate fractions. At lower pH's there were definite differences between the chromatograms of the as-milled flour and those of the fine and intermediate fractions. A major peak present in the original flour was missing from the two fractions and there were lesser differences between the two fractions themselves.

Polyacrylamide gel electrophoresis allowed better resolution than starch gels at the same buffer conditions and avoided an anomaly which appears in the latter type of gel on the anode side. At least 15 electrophoretic components have been found in an aqueous extract (pH 6.0) of a hard red spring wheat flour. Ion exchange separation has been quite effective in fractionation of regular progression, in keeping with sequence of components off the column. Four or five components showing greatest migration toward the cathode were missing after fractionation; they appear to be contained in material passing unadsorbed through the ion-exchange column. Rechromatography has improved the separations, although none of the components have yet been obtained in completely homogeneous condition as determined by gel electrophoresis. The effect of a sulfhydryl blocking reagent (N-ethylmaleimide) on the electrophoretic pattern has been tested and no difference has been noted. Greater fluidity of the extracting mixture containing NEMI was quite evident in keeping with its normal action.

Electrophoretically pure preparations of single components have not yet been prepared in significant quantity, but substantial concentrations of major components are now available for amino acid analysis and tests of functionality. The electrophoretic anomaly or discontinuity appearing in starch gels but not in polyacrylamide gels appears to be due to a definite component which also is the highest in beta-amylase activity. This enzyme activity is eluted from columns over a fairly wide range, but with a definite peaking of activity. It is among the last components to be eluted from the cellulose ion-exchange columns, but is among the constituents moving most rapidly toward the anode during gel electrophoresis, in agreement with earlier observations using paper chromatography.

7. Wheat Germ Proteins. Public Law 480-supported research on the separation and characterization of the major protein and non-protein nitrogenous constituents of wheat germ is just getting underway at the University of Bologna, Bologna, Italy. Knowledge of the free amino acids, nucleic acids, phospholipids, and free amines in wheat germ will provide means of better utilization of wheat fractions for food supplementation and in mixed feeds.

8. Enzyme Action in Low Moisture Grain. Studies of enzyme actions in solid natural products, in relation particularly to water contents in the range occurring in cereal grains, are being carried out under P.L. 480 at the Institut National de la Recherche Agronomique in Paris. Major emphasis is being placed on cereal lipases, and studies will involve kinetic investigations of their activities on enzyme--synthetic substrate mixtures under various well-defined conditions of water vapor pressure, temperature and oxygen partial pressure.

9. Protein Interactions. Basic studies of the interactions among wheat proteins and the contributions of individual components to the properties of the natural protein systems were recently initiated. Preparation of working quantities of purified individual proteins, especially those of gluten, was commenced as a first step toward elucidation of specific interaction mechanisms. Empirical studies of relations between mixing, sulfhydryl groups, and solubility properties of the proteins initiated earlier were intensified.

Comparisons of the amounts of protein extracted from flours and freeze-dried doughs by acetic acid and aluminum lactate buffer demonstrated that the development of doughs by mixing alters the protein constituents in such a way as to increase the extractable protein. For example, 2/3 of the nitrogen of a spring wheat flour was extractable, but 85% could be obtained from the dough mixed to a maximum resistance in a farinograph recording dough mixer. The increase was shown to be at the expense of material which when the original flour is suspended in the dilute acetic acid or buffer, settles rapidly and appears to be highly hydrated.

The rate and extent of conversion to extractable protein in doughs mixed in a farinograph differed markedly among four flours. More vigorous mixing (mixograph recording dough mixer) converted more protein to an extractable form more rapidly than the farinograph. When salt was added to doughs, both the rate and extent of the changes were decreased. In doughs mixed in a nitrogen atmosphere, extractable protein initially increased more rapidly than in air but in 20 minutes (farinograph), the increases in air were larger. In each case the nature of the changes indicated that the conversion of protein to an extractable form was related to the changes in physical properties, as indicated by the recording dough-mixer curves.

A Montana hard red winter flour of very poor mixing stability gave the highest values so far observed for both flour and doughs. A Nebraska hard red winter flour of very good mixing stability gave the lowest. The largest change upon mixing, however, was found with a hard red spring flour.

The addition of a sulfhydryl-blocking reagent, N-ethyl maleimide (NEMI), consistently increased both the rate and extent of change in extractable protein. In general, with this substance present, the maximum increase occurred in 5 to 10 minutes mixing in the farinograph, versus 20 minutes or more in its absence. The maximum proportion of protein extracted was increased an average of 9% by addition of NEMI (from 84 to 93%). Insoluble proteins amounting to about 30% of total flour proteins have been concentrated by high-speed centrifuging of highly hydrated residues remaining after extraction of flour with 0.01 N acetic acid. At 20,000 X gravity, whole starch granules are sedimented out of the gel quite completely. The dried gel fraction contains about 25% protein; insoluble pentosans and starch degradation products undoubtedly are still present.

Results obtained in these studies suggest that the change in acid-extractable protein with mixing occurs more readily and more extensively with spring wheat flours than with winter wheat flours. An interesting lead to further studies is thus provided.

10. Wheat Flour Dough Rheology. Basic studies aimed at developing methods for applying rheological principles to the measurements of properties of wheat flour doughs have very recently been initiated under P.L. 480 in the Rheological Laboratory of the Israel Institute of Technology at Haifa. Early activity under this grant has consisted primarily of assembly and modification of equipment to conduct this research.

11. Gliadin and Gliadin Derivatives. An important part of the program to increase the industrial usage of wheat is the development of high-value food uses for the protein fractions. Work has been concluded during the past year on the development of new and broader food uses for gliadin and deamidized gliadin.

Initial experiments with gliadin and deamidized gliadin as stabilizers in the foam-mat drying of fruit juices produced results at least as good as other proteinaceous stabilizers which are preferred over monoglycerides for fruit juices such as orange and pineapple. Regular gliadin gave better results than the deamidized gliadin. In cooperative studies with a local beverage producer, gliadins were evaluated as a foam-producer and stabilizer. It was found that gliadins have good foaming power but also cause serious haze formation upon chilling and/or storage. The pepsin-hydrolyzed gliadin performed best but haze formation with all three types of gliadin was heavy. This defect has so far been found with all proteinaceous foam stabilizers yet investigated and probably results from reaction with tannin systems present in the beverage. The high amide nitrogen content of gliadins is probably responsible for its affinity for tannin substances; nylon (also high in amide nitrogen), for example, can be used to remove the tannins from these liquids.

A simplified laboratory procedure was worked out for deamidation of gliadin by direct treatment of precipitated gliadin with hydrochloric acid thus avoiding the dilutions to 5 to 10% solutions previously used. Gliadin phosphate was prepared by heating a mixture of dry gliadin, phosphoric acid and urea. Solubility properties of the phosphorylated gliadin appeared generally similar to those of partially deamidized gliadin.

In further studies designed to reduce costs of extraction, acetic acid was substituted for the aqueous alcohol (isopropyl) previously used. By partial neutralization most of the non-gliadin constituents were removed. Complete neutralization then precipitated the extracted gliadin and after washing with water the product contained as little carbohydrate and lipid as gliadins prepared by the earlier procedure. Thus far, foaming properties of the product both before and after deamidizing have been poor.

The glutenin residue after extraction of gliadin with acetic acid, was added to a commercial flour of mediocre bread-baking qualities. Mixing tolerance of doughs and loaf volumes were increased in contrast to the lack of effect of glutenin obtained by the isopropanol extraction procedure.

In studies aimed at simplifying the deamidation of gliadin, and decreasing the cost of preparing deamidized gliadin, it was shown that amide groups could also be removed by autoclaving a gliadin mass wet with acid which would increase the capacity of equipment for such a process. Sulfuric acid yielded products difficult to dissolve at neutral pH. Hydrochloric acid presents problems in corrosion of the autoclave and in "boiling over" of the gliadin mass during treatment. When phosphoric acid was used it was found that the gliadin phosphate was soluble at neutral pH, insoluble at pH 4 and had good foaming properties. It thus appeared to be simpler to use phosphoric acid to partially deamidize gliadin.

The foaming properties of gliadin extracted by acetic acid from gluten were only slightly improved by treatments with alcohol to remove residual lipids. Foaming properties of gliadin were improved by heating the gluten dispersion in acetic acid prior to separation of the gliadin; extraction with 95% ethyl alcohol then gave a product with foaming properties approaching those of gliadin isolated by the isopropyl alcohol method.

Ethyl and glycerol esters of gliadin were prepared by reaction of the alcohols with gliadin under anhydrous conditions in the presence of HCl. The esters had solubility properties similar to those of gliadin rather than deamidized gliadin; they were soluble at slightly acid pH, insoluble at neutral pH, and easily precipitated by salt. The ethyl ester gave poor foams; the glycerol ester gave

much better foams, but not as good as unmodified gliadin. Attempts to prepare a glucose-gliadin ester were not successful.

Beneficial effects on foaming properties of gliadin prepared from heated acetic acid dispersions of gluten appear to result from inactivation of proteolytic activity in the gluten. Viscosity decreases occurring in the absence of heating were accompanied by an increase in free amino groups but no increase in ammonia. Possible contaminating microorganisms were eliminated as a cause of the viscosity decreases. Heating in glass containers produced less denaturation of gluten protein than heating in stainless steel containers. Freeze-drying of reaction mixtures in the preparation of gluten and gliadin phosphates produced products as good or better than those obtained by the tedious and prolonged air-drying step formerly used.

Completion of these studies during the past year has allowed placing of added emphasis on other new products research.

12. Ultrasonic Study of Wheat Gluten. Studies of alterations in the chemical and physical properties of wheat gluten induced by ultrasonic vibrations are being carried out under P.L. 480 at the Institut National de la Recherche Agronomique in Paris. In early studies, it has been shown that ultrasonic treatment of gluten causes a decrease in solubility and viscosity, the latter being more pronounced than expected from solubility loss.

13. Phosphorus in Wheat Flour. Basic studies on the effects of phosphorus compounds on the baking qualities of wheat are being carried out under P.L. 480 at the Institut National de la Recherche Agronomique in Paris. Ten U.S. wheat samples, supplied by the Western Utilization Research and Development Division, are being evaluated. The amounts of phospholipids, phosphoproteins and phytin present in flours of known baking character are being determined.

14. Solubility of Gluten Proteins. Research on the solubility of wheat gluten proteins is being conducted under P.L. 480 at the Centre National de la Recherche Scientifique, in Montpellier, France. These studies are providing a better knowledge of the characteristics of gluten proteins in relation to their foaming and surface-active properties and of the means of increasing and decreasing the solubilization of these proteins in neutral solutions. It is expected that the results of these researches will provide the basis for developing means for improving the baking properties of flours, and will assist in the selection of wheats most suitable for specific uses.

15. Flavor Studies. Current trends indicate that abbreviated and continuous-mix baking processes with their marked economic advantages

will be used more and more by the baking industry. In these processes, pre-ferments must be used to provide a major part of the flavor derived from fermentation. The objective of this research is to gain information which will permit control of the pre-ferment process so as to make continuous process bread equal, or perhaps even superior to conventional, more expensive bread.

It was reported previously that twelve of the more than the twenty volatile organic acids produced in pre-ferments had been identified on the basis of retention volumes on a gas chromatographic column. During the past year six additional acids have been identified. This was accomplished by utilizing the emergence times of the free acids and of their ethyl esters (the acids are formic, lauric, myristic, crotonic, palmitic, and pyruvic).

Decreases of 5° and 10° C. in the temperature at which pre-ferments are prepared, had no effect on the total concentrations of acid and alcohol produced, but concentrations of both volatile and non-volatile carbonyl compounds declined proportionately to the temperature level used. The amounts of carbonyl compounds in pre-ferment show an interesting and consistent trend as fermentation proceeds. Volatile carbonyls increased steadily in amount throughout the six-hour reaction time. Non-volatile carbonyls, however, increased to a maximum within two to four hours and then decreased substantially. This effect is more prominent in pre-ferments made with relatively high sugar concentration. The non-volatile carbonyls appear to consist predominately of aldehydes and/or keto acids such as pyruvic acid, mentioned above.

In further work the separation of organic acids from pre-ferments prior to examination by gas chromatography has received attention. Extraction with methylene chloride gave reproducible results but it extracted different acids to different degrees, e.g., capric acid over 50%, acetic, lactic and pyruvic, about 5%. Little if any unsaturation was present in the extracted material. For quantitative analysis the procedure is not satisfactory. Therefore attention is now being given to the separation of acidic components from the pre-ferment mixtures by ion-exchange.

A method employing column chromatography for determination of total organic acids had been used earlier for analysis of pre-ferments. Its applicability to doughs was investigated. High recoveries of pyruvic, lactic, and acetic acid added to doughs was obtained.

Total organic acids were shown to increase moderately in concentration during the proofing (pan fermentation) of bread doughs made by a pre-ferment procedure; baking caused a larger increase. Total carbonyl compounds and total alcohols follow a similar course, except that the alcohols and volatile carbonyls drop to very low

levels as a result of baking. Among the volatile organic acids produced during fermentation, formic and alpha-methyl butanoic acids have now been clearly identified. This brings the total identified to 18. Gas chromatography of all acids present as their esters indicate the presence of at least 45 different components, although this total would probably include mono- as well as di-esters of dicarboxylic (non-volatile) acids present. Chemically leavened (glucono-delta-lactone plus bicarbonate) breads prepared with two- to four-fold concentrates of pre-ferments were at least equal, if not superior, in aroma to breads made by a typical laboratory sponge and dough method.

Aqueous isopropyl alcohol has been found to be a more effective solvent for carbonyl compounds in bread and bread doughs than water alone. Analyses of freshly mixed bread doughs, proofed doughs, and breads baked from the doughs have shown that the greater the starting concentrations of sugar and yeast in the pre-ferments used, the greater the concentrations of flavor substances in the dough and breads, as would be expected. Alcohol production during pan-proof is greater in doughs from richer pre-ferments, but production of total acids and total carbonyls is unaffected by starting levels of sugar and yeast. Preliminary taste panel tests indicate that breads made with the richer pre-ferments have more pronounced flavor and aroma, in keeping with the chemical results obtained and validating the practical importance of the work.

Preliminary baking experiments with chemically leavened doughs indicate that higher flavor-potency concentrates are obtained by freeze-concentration of pre-ferments containing yeast cells than from pre-ferment supernatants. Freeze-concentrated pre-ferments of either kind are more effective sources of fermentation flavor precursors for bread than any other experimental or commercial flavor-enhancing substances yet investigated. Fractionation of concentrates into neutral, acidic, and amino acid fractions has been undertaken to locate the source of the bread flavor precursors furnished by pre-ferments.

In studies of flavor as related to baking procedures, research was conducted to test the validity of the hypothesis that the formation of the brown coloration and attendant unique aromas and flavors in the crust of freshly baked bread is based on the reaction of amino compounds and reducing sugars (by way of the Maillard reaction, followed by the Amadori rearrangement). Crystalline products from glycine and glucose were prepared for model system study of the proposed reaction mechanisms. These products exhibited none of the fluorescence that eventually occurs as the browning reactions proceed, and which can be extracted from bread. It was noted that no fluorescence develops in an aqueous solution of the crystalline Amadori product during six weeks at room temperature or after one

hour at 100° C. Continued heating produced gradual browning and slowly-developing weak fluorescence. Increased fluorescence and crust coloration resulted from addition of 0.5% glycine (based on weight of flour used) to chemically leavened and yeast leavened bread. The fluorescence extractable from bread was almost completely confined to the crust. Based on these results it is presumed that all final Maillard reaction products arise from simple decomposition of Amadori compounds. The slowness of the development of fluorescence in browning during heating of the glycine-glucose Amadori product suggests a strong possibility that other reaction pathways are also important in the browning of amino acids and reducing sugar.

Seventeen free amino acids have been identified in fermented bread doughs ready for baking. In aggregate the free amino acids appear to account for the major part of the dialyzable amino-group-containing substances present in fermented doughs. Peptides of medium to low molecular weight appear to be absent or present in only very small amounts. In test tube experiments, browning induced between a number of amino acids present in doughs and several aldehydes produced during fermentation failed to produce any odor at all reminiscent of baked bread. Gliadin and gluten likewise failed to produce pleasant odors. Gas chromatograms obtained by very sensitive hydrogen-flame ionization equipment (aromagrams) from vapors of freshly baked bread crust and crumb differ markedly.

Quantitative studies have shown that up to 90% of the free amino groups available in fermented bread doughs for reaction with reducing carbonyl compounds in crust browning are furnished by insoluble proteins. Free amino acids and water-soluble proteins contribute less than 5% each. Ammonium ion concentrations also are low in magnitude. Pre-ferments used for the bread doughs also contain small amounts of free amino acids, ten of which were identified.

Improvements in gas chromatographic techniques have produced linear relationships between sample concentrations of organic acids and peak heights obtained with capillary chromatographic columns. Sample injection precision of $\pm$ 2 to 3% has now been obtained, and sensitivity of detection of individual compounds has been increased sufficiently to permit reliable detection of micromicrogram quantities of materials. Quantitative estimation of individual flavor compounds can now be undertaken with good confidence.

16. Pre-Ferments in Commercial Bread Baking Procedures. Contract research is underway at the Massachusetts Institute of Technology with the objective of determining the effect of modifying the composition of commercial-type pre-ferment doughs upon volatile flavor and aroma components in bread. Information gained in this work should aid in developing methods for producing commercial bread with enhanced flavor.

In early work, bread of suitably high quality and batch-to-batch uniformity was prepared by rigorous control of operating variables particularly a standardized time schedule. Flavor concentrates were prepared by vacuum distillation of volatiles from fresh bread into a series of suitable cold traps from which they were recovered by thawing under nitrogen, saturating with salt, and extracting with ethyl ether. The condensed liquid in the traps exhibited a typical fresh bread odor when thawed.

Analytical work has included the initiation of a survey of gas chromatographic column packings, conditions of operation, and methods for trapping and rechromatographing fractions. Initial results show that at least nine major components (including ethanol, acetone, diacetyl and crotonaldehyde) are detectable using column chromatography. Standardization and stabilization of the organoleptic taste panel methods has been accomplished. Triangle tests were selected as being most suitable and were used to show that 500 p.p.m. of L-leucine added to doughs produced a significant change in the flavor and aroma of the resulting bread. Quantities of flavor and aroma material and chromatograms (thermal conductivity detectors) for the recovered material were satisfactorily duplicated in separate extraction trials on different lots of fresh bread. The effect of addition of six individual amino acids, gliadin, casein, lactalbumin, maltose, and lactose to doughs has been evaluated organoleptically in triangle tests on bread crust and bread crumb. No difference was found between the effects of L-isomers or DL-mixtures of amino acids. A number of significant differences in odor or taste of crust or crumb resulted from the formula additions, but in most cases the control bread was preferred by the taste panel. The notable exception was proline. The odor of crumb and the odor and taste of crust of bread containing added proline were preferred by the panel over controls. Bread containing added proline was therefore selected as the subject for quantitative chemical examination of flavor and aroma components. Of the 16 compounds detected in flavor isolates, eight are major components, and seven of these have been rigorously identified. The remaining compounds are minor in amount, but tentative identity has been assigned to five. Of the carbonyl compounds present in the concentrates, acetoin and furfural are much more prominent than any others. Alcohols of detectable and measurable quantities include ethanol, propanol, isopropanol, and isobutanol. The lesser components present in flavor isolates appear to occur in quantities of the order of 3 to 6 parts per million.

B. New and Improved Food Products

1. Bulgur, the Parboiled Wheat Food. The development of canned whole-grain ready-to-eat bulgur products especially suited for the domestic market as a convenience food, but also for possible use in

the export trade has been reported previously. In the past year, there has been considerable outside interest in this new wheat food product, including a market test sponsored by a Midwestern State wheat commission. Research has continued towards enhancing the convenience features of the product.

A sample of hard red winter wheat from the intermountain region was found to take up water much more rapidly during pilot-scale processing than the club wheat used in previous studies, both for the production of canned whole-grain bulgur and the seasoned bulgur, called pilaf. The peeled red wheat kernels, however, were much less prone to disintegrate during the high temperature cooking phases and gave quite a satisfactory texture in the final product. This resistance to overcooking is probably a function of the higher protein content of the red wheat as compared to the club wheats which are normally quite low in protein.

Discrimination and acceptability tests conducted by the Agricultural Marketing Service with our cooperation on whole-grain and cracked grain pilafs prepared at two levels of seasoning from the hard red winter wheat showed that untrained tasters could not reliably distinguish between the two levels of seasonings used. Acceptability by untrained tasters totally unfamiliar with the products was moderate, a rating received rather frequently for a new product which later becomes well received. The full kernel form of the product received a small edge in preference.

The effect of moisture content of conventional dry bulgur on the degree to which it expands during a hot-air puffing operation was determined. Puffed volume increased regularly as moisture content decreased from 14% to 8%, consequently, the behavior of bulgur in this respect with a maximum at or below 8% moisture is different than that of rice. In later studies optimum conditions for heat-expanding bulgur have been found to be 500-600°F. air temperature, 400-600 f.p.m. air velocity, 7 to 11% moisture, and bran removal equivalent to about 40% reduction in crude fiber content of the bulgur. Of these variables, only degree of debranning showed any effect on rehydration properties of the puffed material. Instant bulgurs prepared by heat-puffing are suitable for use in dry dessert mixes, dry soup mixtures, salad mixtures, and the like. Commercial formulas for pineapple, butterscotch, and chocolate flavored Bavarian puddings have been developed and are undergoing storage stability tests. Mild toasting of the bulgur for the butterscotch and chocolate puddings is desirable, but not for the pineapple pudding.

Commercial formulations and processing procedures for eight new canned bulgur products are virtually completed. Whole-kernel bulgur was found to be a better starting material for such products than

peeled raw wheat, especially if the products are unflavored or mildly flavored. Special precautions were found necessary for control of kernel texture in canned products containing sugar or acidic materials such as tomato puree.

More than twenty common bleaching agents have been screened for their effectiveness in whitening partially debranned red wheats in development of bulgur-like products of lighter color and blander flavors for overseas markets. Heat treatment of raw wheat prior to bleaching markedly reduces cooking time of the final product. The bleached wheat has been given the name "wheat pearls."

Engineering studies have been carried out in two areas, continuous bulgur production processes, and wheat peeling. In these investigations, pilot-plant studies have led to development of a simple continuous process for production of dried precooked wheat (or bulgur). In this process, cleaned wheat is soaked and precooked in four simple conveyor-type units connected in series. In the first two conveyor units, the wheat is soaked for a total time of about 60 minutes in water ranging in temperature from 140° to 180° F. The grain is then drained and held hot in the third conveyor unit for a 30-minute tempering period. The tempered grain is fed to a wire-mesh conveyor belt on which it is cooked in steam at atmospheric pressure for 15 minutes. The cooked grain is then dried by conventional means. Studies to determine optimum conditions for applying this simplified process to different types of wheat are in progress.

In the area of wheat peeling, laboratory and pilot-scale studies are being made to determine the effects of processing variables in debranning of raw wheat. These studies involve use of milling equipment of the type used in processing of rice and include milling of red and white wheats with and without the addition of water and mild abrasives. Considerable information has been obtained on conditions required to obtain different degrees of debranning. With either moisture conditioning or addition of mild abrasives, or both, more bran can be removed than by either wet scouring or dry milling techniques previously employed. A concomitant unexpected advantage is that much of the germ is also removed by these techniques, a result never before accomplished without subdividing the kernel. A procedure has been found that effects essentially complete removal of colored bran layers from red wheat, but the milling times and weight losses are as yet undesirably high.

2. New Food Products from Gluten. In early exploratory work it was shown that textures and flavors satisfactorily similar to meat products can be achieved by appropriate combinations of wheat gluten, high-protein wheat flours, cracked bulgur, and various appropriate seasoning materials derived from either or both vegetable and animal

protein hydrolysates and extracts. Commercially available wheat gluten, obtained almost entirely from second-clear flours, was found to be too dark and too strongly flavored to be suitable for products simulating veal or chicken products. Glutens washed from baker's patent flours, however, are quite satisfactorily light in color and bland in flavor. Texture modifications have been obtained by appropriate partial denaturation of gluten before incorporation into products and/or by blending with flour or with cracked gluten of differing size-grades. Combinations of appropriate commercially available flavoring materials have been devised to simulate closely, beef-, chicken-, veal-, and fish-flavored products.

Additional work with handmade meat-like combinations based on gluten has shown that texture modifications of formulations resembling loaves, patties, or chopped steaks can be obtained by manipulation of moisture levels and proportions of cracked bulgur or high-protein wheat flour blended with the gluten. Preliminary trials with canned products, such as a vegetable stew containing beef-like chunks of gluten, indicated that no special difficulties need be expected except that texture control may need extra attention. The formulated products also freeze well provided waxy rice or waxy corn starch comprises part of the thickener used for sauces or gravies.

3. Biological Value of Processed Wheat. Studies aimed at developing rapid chemical methods for assay in the biological value of proteins during processing of wheat food products, is being conducted under P.L. 480 at the University of Cambridge, Cambridge, England. Results to date show significant apparent losses in amino acids other than lysine when proteins are heated at moderate temperatures. Excellent results are being obtained in this very timely research which will guide further research aimed at developing foods to alleviate nutritional inadequacies around the world.

4. Products from Air-Classified Flours. In cooperative studies with the Northern Utilization Research and Development Division, baking evaluations and soluble protein analyses were conducted on 15 samples of flour derived by fine grinding and air-classification of pastry flour commercially milled from the Pacific Northwest club wheat. Three blends containing 11.5 to 12.0% protein produced very poor bread. A low protein hard red winter wheat flour enriched with a high-protein air-classified club wheat fraction also produced very poor bread. Six of the seven blends of the air-classified fractions suitable for cake flours, produced good white cakes after bleaching; the seventh, a coarse residue fraction, produced a fair cake. The reground coarse residue, however, produced good cakes. The flour blends designed for cookies produced very good, good, fair, and very poor cookies. The contents of total soluble proteins,

total albumin, and total globulin in the original flour were consistent with results obtained previously for flours of a similar type. Protein shifts caused by air-classification did not alter the characteristically low albumin/globulin ratio of the original flour, although the percentages of the soluble proteins varied inversely, but moderately, with the total protein content of the fractions obtained.

In extending these studies to other wheats, flours milled from the varieties Brevor, Burt, Rio, and Omar were fractionated by air classification at the Northern Regional Research Laboratory and evaluated at the Western Regional Research Laboratory. Fine fractions containing 15 to 26% protein were combined with each of 3 medium-protein flours (9.7, 10.3 and 10.7% protein) to give blends containing 12% protein. Bread loaf volume and score were increased most by the Brevor fractions, followed by Rio; they were not changed by Omar fractions and were decreased by Burt fractions. Water absorption was markedly higher (6.2% over-all average) for the blends than for the base flours. Burt fractions gave the largest increases, followed by Rio; the increases parallel starch granule damage. Stability of the blends to mixing in the Farinograph was only slightly changed.

Better cookies were obtained from Brevor and Omar samples than from Rio and Burt. Within varieties, however, marked differences were found in which granulation must be a factor. Thus regrinding coarse fractions or the original flours resulted in large decreases in cookie spread. Nevertheless, there was no definite limit in particle size below which spread was poor, and fine fractions from the Brevor wheat gave high spread values.

Cakes were baked (after chlorine bleaching to pH 5) from low-protein fractions. With the methods used, no obvious trends were found. In general, however, good cakes were not obtained with coarse fractions that gave viscous batters.

5. Wheat Flour Lipids. The effects upon baking quality of composition variations in wheat flour lipids is being investigated under P.L. 480 at the British Baking Industries Research Association in Chorleywood, Herts, England. The effect upon baking quality of added fat, of location of growing of wheat, and effect of nitrogen fertilization are all being investigated. Thin-layer chromatography is being utilized to compare lipids in the flour, dough, and bread. The U.S. wheats under investigation are being supplied by the Western Utilization Research and Development Division.

6. Food Supply in Fallout Shelters. Investigations on processed foods suitable for provisioning fallout shelters are being conducted using funds transferred to Agriculture by the Department of Defense.

Two lines of research are involved; (1) Extension of research on the bulgur-type wheat wafer previously conceived by the Western Utilization Research and Development Division for stockpiling in fallout shelters, and (2) development of a line of adjuncts for use with the bulgur-type wheat wafer.

Wheat bulgur wafers are composed of about 80% puffed bulgur, 10% shortening, 10% malt sirup solids and a small quantity of salt. The ingredients are warmed, mixed, and pressed in a die using several tons pressure. The protein content of the wafer is fixed at 7.5 to 8.5%, a level established as the optimum for situations where water intake is limited.

From information obtained from experimental production on a laboratory scale, a commercial-scale production line has been proposed and cost estimates prepared. Several variations in wafer formulations and methods of preparation have been investigated. Some of the changes in formulation are included in the storage stability studies to be conducted under the contract discussed below. Cookie-forming equipment, extrusion presses and similar equipment using dampened mixes were found to produce wafers substantially lower in density than those formed by pressing.

To determine the effectiveness of changes in formulation on relative stability, accelerated test methods were used. The Oxygen Bomb Stability Apparatus failed to produce usable results. A new approach has been made toward an accelerated test method for bulgur wheat wafers by employing gas-liquid chromatography (GLC). Combining the oxygen bomb for treatment of the sample and GLC for measuring changes gave promising results in preliminary tests. For example, GLC measurement of hexanal increased in samples treated in the oxygen bomb for increasing time periods. Similarly, the samples protected with increasing amounts of antioxidants showed decreasing amounts of hexanal for equal treatment in the oxygen bomb.

A research program for evaluating the stability of wafers by taste panel methods on samples stored at three temperatures, 40, 70 and 100° F. over a five year period was developed and initiated by contract with Oregon State University. Sixteen different formulation package combinations will be studied representing a complete factorial of four variables, (1) red and white wheat, (2) pressure cooked and atmospheric cooked bulgur, (3) malt sirup and corn sirup binder, and (4) air and nitrogen atmospheres in the package. Simultaneously chemical analyses will be performed, selected on the basis of their usefulness in measuring changes which may be related to wafer quality and useful in predicting shelf life of wafers.

The wafers offer unusual versatility in use. It may be eaten out of hand, plain, or with spreads or icings and it may be crumbled

for use with soups, sauces, gravies, and toppings to provide a diversity of dishes. A number of adjuncts for use with the wafer have been developed. These are dehydrated mixes that may be readily reconstituted for use. Preliminary formulations for nearly 60 different foods were developed. Evaluations and modification of formulas are to continue.

C. New and Improved Food Processing Technology

See, under paragraphs B, 1 through 6, product development work that is related to improved processing technology.

D. New and Improved Feeds

1. Estrogens in Wheat Bran and Germ. Contract research is underway at the State University of Iowa, Ames, Iowa, to determine the chemical nature of potentially estrogenic compounds that can be isolated from wheat germ and bran as a basis for the development of products of enhanced value in the fattening of livestock and poultry. This work is a part of a fundamental approach to upgrading non-flour fractions of wheat. These fractions which comprise about 30% of domestically-milled wheat have been sold conventionally as feed components. Markets were lost in feeds for non-ruminant animals due to the current trend to higher energy in this type of feeds. Development of specific uses due to biologically active components can be expected to increase their dollar value and thus increase the overall value of the farmer's product.

In earlier work the ether-extractable non-saponifiable constituents of wheat germ were fractionated into four major groups by chromatography on alumina columns. These groups were: a) a hydrocarbon fraction, b) a carotenoid fraction, c) a high-melting steroid (m.p. 160-165° C.) fraction, and d) a "beta-sitosterol" fraction (m.p. 130-140° C.). Gas chromatography of the hydrocarbon fraction revealed nine major and almost twenty minor peaks. Similarly, four peaks not well separated were found in the steroid fraction. Bioassays of the steroid fraction and of two arbitrarily selected portions of the "beta-sitosterol" fraction showed no estrogen-like activity.

The non-saponifiable constituents of bran oil were separated into five fractions: a) a hydrocarbon fraction, b) a "middle" fraction, c) a steroid fraction, d) substance A (crystalline), and e) an end fraction. Refractionation of the middle fraction by chromatography on alumina separated it into four fractions: 1 and 2 were oils of distinct infrared absorption patterns, 3 substance A, and 4 "beta-sitosterol" fraction (m.p. 130-138° C.). Fractionation of the bran non-saponifiables on the basis of alcohol solubility and refractionation by chromatography on alumina leaves small amounts of substance A in most of the other fractions. It can, however, be

recovered from the other fractions by use of a special extraction procedure employing Claisen's alkali and petroleum ether. Preliminary bioassays indicate that both substance A and the hydrocarbon fraction possess estrogen-like activity. Initial attempts have been undertaken to determine the structure of substance A. A preparation showing only one spot on thin-layer chromatography analyzed $C_{14}H_{26}O$ (or $C_{28}H_{52}O_2$). An observed molecular weight of 437 compares well with the weight of 421, calculated for the dimer. Infrared and ultraviolet absorption spectra have been obtained on the original molecule and its crystalline acetate. Nuclear magnetic resonance spectra have also been obtained on the acetate derivative. Elemental analyses on the compound and its acetate limit the forms to $C_{25}H_{44}O_2$ or $C_{26}H_{46}O_2$ formulas. This suggests it to be a long-chain alkyldihydroxybenzene. Crystallization of the almost pure acetate derivative from methanol reverted it to mixtures. Thin-layer chromatography revealed that relatively rapid solvolysis was taking place and that the mixture contained the original acetate, an intermediate substance, and substance A. Furthermore, it could be shown that both acetylation of the latter and hydrolysis of the original acetate leads through a single intermediate--the first indication that a dihydric phenol of symmetrical structure might be involved. These data, the nuclear-magnetic resonance data, and a reasonable biosynthetic assumption of substance A being of polyacetate origin form possible structures differing only in the symmetrical ortho or meta position of the two hydroxyl groups to the long alkyl chain having either 19 or 20 carbon atoms. Ozonolysis of substance A has yielded an acid, crude m.p. 68-72°. While this compound is still in a crude state, it may be of significance in that its melting point is in the range close to those of stearic (C_{18}) arachidic (C_{20}) and behenic (C_{22}) acids.

E. New and Improved Feed Processing Technology

1. Upgrading Wheat and Barley Feeds. It had been demonstrated earlier that nutritional properties of grains can be markedly improved by laboratory-scale processing treatments with water and with enzymes. Basic knowledge was lacking, however, of the chemical changes occurring in these grains during these new treatments. Such information is essential for development of commercially feasible processes. Contract research in this area has been recently completed at Washington State University, Pullman, Washington. Based on this study it appears that economical methods can be developed for treating western-grown cereal grains and products derived from them in order to obtain their maximum feed value in high-energy growth rations for chicks, and further, to discover the mechanisms by which the improvements are obtained. In the work reported previously it was noted that the largest part of the positive effect of water treatment of barley and wheat and their fractions was due to combined effect of enzymes, and antibiotics produced by

microorganisms of the B. Subtilis type, which grew during the slow-drying process used on the wetted grains. More recent results are not completely in agreement on this point. For chicks, the weight of evidence is still in this direction. For turkey poults, however, newer results have shown that water treatment gives a greater effect than bacitracin plus fungal amylase. Furthermore, an additional response was obtained by feeding terramycin in either case. It is noted also that in chicks, wheat bran was improved more by water treatment than by the antibiotic, bacitracin, plus fungal amylase supplementation. The importance of microorganisms in the barley water treatment on the effect on growth was clearly demonstrated by experiments utilizing ethylene oxide sterilization. An additional important point that was clarified by these studies was the following: feeding barley was found to cause hypertrophy of the pancreas but this effect was reduced by water treatment of the barley.

Very little effort was possible "in-house" because of efforts expended on other high priority research. It was demonstrated, however, that supplementation with commercial enzyme mixtures and zinc bacitracin of high-energy broiler rations containing various wheat fractions failed to overcome the small but significant growth inefficiencies previously observed in chicks fed rations high in the endosperm fraction of wheat. Growth rates on diets containing about 48% of second middlings or second low-grade flour stocks were as low or lower than those for diets containing 30% of bran or germ. Much of the growth diminution obtained on the latter diet is directly attributable to their high fiber content. Responses to the enzymes plus antibiotic additions were either negative or only very slightly positive on all diets. The greatest response was obtained on the whole wheat diet.

The results obtained contrast with those found for barley on which starch digestibility and availability are improved as much by the enzyme plus antibiotic additions, as by water treatment. Thus the possibility still exists that some deleterious substance is present in wheat endosperm, although water treatment of such fractions must still be investigated before the digestibility hypothesis can be eliminated with confidence.

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Area 2 RICE--PROCESSING AND PRODUCTS--WESTERN LABORATORY

Problem. The productive capacity of U.S. rice growers has increased faster than domestic and export consumption over the past decade, thus limiting the income potential from this major world food grain. Detailed knowledge of chemical composition and physical properties, as related to processing is needed to guide milling, processing, and product development of U.S. rices so that they can better meet the quality requirements necessary for expanded markets. New and diverse food products from rice that are easy to prepare, have flavor and texture appeal, and are economical to manufacture, are needed to increase the total consumption of rice both domestically and abroad.

USDA PROGRAM

In the Western Utilization Research and Development Division, basic and applied research on rice is conducted at the Division headquarters, the Western Regional Research Laboratory in Albany, California. Basic studies involve chemical, physical, and biochemical investigations of rice proteins and amylase enzymes. The protein work is concerned specifically with isolation and characterization of the globulin and glutelin proteins of the endosperm. Applied research underway is aimed at developing improved white and brown rice products by studies on the gelatinization of starch during parboiling. Preparation of high-protein rice fractions by means of fine-grinding and air-classification techniques is under exploratory investigation.

The Federal program of research in this area totals 3.8 professional man-years. Of this number, 1.3 are assigned to chemical composition and physical properties; 2.0 to new and improved food products; and 0.5 to new and improved processing technology.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 1.5 professional man-years of effort on rice. Of this number, 1.2 were devoted to chemical composition and physical properties, and 0.3 on new and improved food products. The compositional studies are concerned with extraction of rice proteins using detergents, and the properties of the isolated proteins. Product research is concerned with cooking, milling and processing characteristics of rice varieties and selections, and the effect of environmental and related factors on these characteristics.

Industry and other organizations including food processors, milling companies and grower organizations conduct research programs that are predominantely concerned with specific applications to individual

corporate problems. Three or four milling companies are investigating product improvement and development. Information gained is largely confidential or patented. Limited applied research is being conducted by breakfast food and baby food manufacturers, and brewing companies. Total effort in this area by industry is estimated as being equivalent to approximately 10 professional man-years per year. Grower organizations cooperate with government laboratories in the supply of samples. The level of effort has always been less than one man-year equivalent.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Rice Proteins. A method has been developed for isolating globulin and glutelin proteins using dilute salt or alkali. Purification of these isolates gave two globulins and one glutelin. The glutelin was found to be very difficultly soluble. The physical properties of these protein preparations were determined. The globulin proteins were examined by starch gel electrophoresis. At least ten different components were shown to be present. The demonstrated applicability of this method of analysis will hasten progress in research on rice proteins. These compositional investigations will be emphasized in continuing studies.

B. New and Improved Food Products

1. Parboiled Rice Foods. Limited studies have been conducted on the parboiling of rice to answer specific questions which arose in connection with the production of parboiled rice for export to India.

Preferred conditions for parboiling Calrose rice were determined including optimum steeping, tempering, autoclaving, and drying conditions. It was shown that tempering reduced the amount of water that had to be removed during drying. A few tests indicated that vacuum treatment prior to or during steeping hastened steeping and reduced the percentage of broken kernels in the milled rice. It was also shown that when the rice was steeped at temperatures above that required for gelatinization, the product became sticky and clumped badly. These findings led to added emphasis being placed on studies of gelatinization.

It was demonstrated that the degree of gelatinization by steam treatment during preparation of quick-cooking and instant products, can be determined and controlled by following changes in hot-paste viscosity characteristics. The peak viscosity in Amylograph curves and the viscosity after 20 minutes at 95° C., show a regular change in relationship with one another as gelatinization progresses in the steam treatments of the grain. The large initial differences in the two

curve values diminish steadily as gelatinization progresses. The treated rice appears to be fully gelatinized when the difference in curve values reaches zero. Moisture content of the rice during steaming appears to be the principal factor limiting rate of gelatinization, whereas steaming time governs the degree of gelatinization irrespective of moisture, within practical limits. Tempering time prior to steaming has no effect on rate or extent of gelatinization.

The Amylograph method for determining degree of gelatinization has been shown to be equally applicable to brown rice, although small differences in curves for the two types of rice were noted. Absolute values for peak viscosity, and viscosity after 20 minutes at 95° C. are lower for brown rice; and rates of change in peak values, as a result of processing, differ somewhat.

Completion of the aforementioned studies has made possible a shift in effort to expand investigations on the relation of protein composition to processing properties.

2. High-protein Rice Foods. In cooperative studies with the Rice Experiment Station at Biggs, California, rice of high-protein content has been produced on soil with high nitrogen fertilization. The rice had a protein content of 10%. Studies on the cooking and processing qualities of these rices have been initiated. Interest in these rices is high because of their possible use in bland high-protein food products for children.

C. New and Improved Processing Technology

1. Rice Drying. In earlier studies, procedures were developed which enabled dryers of western short-grain rice to find the most effective operating conditions to assure more unbroken rice, increased drying plant capacity and reduced drying costs. Rice dried to moisture levels lower than those ordinarily obtained in conventional drying establishments showed increased yields of head rice. The greater returns obtainable by virtue of the smaller loss from breakage, however, would be overbalanced by the weight shrinkage unless the moisture loss was restored by rehydration. Studies on the applicability of these results to southern long-grain and medium-grain rices have now been successfully completed in cooperation with scientists of the Southern Utilization Research and Development Division. Certain modifications in sampling techniques and methods of determining milling quality of dried products were found necessary in order to obtain reliable results. These studies were conducted in the plants of cooperating rice processors. No further studies in this field are anticipated for the present.

2. Rice Preprocessing Behavior. Knowledge of the effects of preprocessing history to processing behavior of rice is important to guide production of rice of a particular variety with the most desirable

properties. The major part of the investigation under this project was performed on 1) samples of three western-rice varieties (Caloro, Calrose and Colusa) harvested at successive stages of maturity and 2) samples of Caloro and Calrose rices grown under different applications of nitrogen and phosphorus fertilizer to the soil, and harvested when fully matured. These studies were conducted cooperatively with scientists at the Rice Experiment Station, Biggs, California.

Properties of the samples evaluated in the maturity series and fertilizer plot series were kernel weight, total and head rice yields, chemical composition, hot paste viscosity of flours, water adsorption at different temperatures, color components, and chalkiness. Factors other than cultural practices studied with reference to the viscosity of rice pastes were variety, amylase content, amylolytic enzymes, and holding at different temperatures subsequent to milling.

A major finding of this study was that the numerical values for various rice properties attained maxima or minima during maturation at about the same point which, in most instances, was within the range of 25 to 30% harvest moisture; the properties being head yields, water adsorption, and color components. Some composition factor doubtless accounts for this behavior pattern. Its identification will require a more detailed study than was undertaken.

3. World Rice Evaluation. Cooperative research on world rices has been completed, the cooperation involving Foreign Agricultural Service, Agricultural Marketing Service, and other Agricultural Research Service agencies of the USDA. In this study, a total of 264 samples of rice, mostly short-grained types, were analyzed, in part at WU, together with 16 reference samples of domestic rice varieties. The majority of samples received were analyzed for moisture, Kjeldahl nitrogen, surface lipids, total starch, and amylose, by methods standardized in advance by the cooperators. Color characteristics were determined by the Hunter color and color difference meter. Selected samples from groups of the particular variety were further analyzed for total lipids, crude fiber, and ash. Amylose:amylopectin ratios were calculated for all samples. No prominent chemical differences among the short-grained foreign rices and our domestic varieties were found in preliminary examination of the data. The general appearance of the foreign rices was decidedly inferior to U.S. rices.

These studies are complete; further work in this field is not contemplated.

4. Air-classification of High-protein Rices. Cooperative work with the Rice Experiment Station, Biggs, California, on the growing of high-protein rice through use of high nitrogen fertilization, is also reported above. Studies have shown that application of 80 pounds of nitrogen per acre increases the nitrogen content of rice by as much

as 50%. No change occurs in the ratio of non-protein to protein nitrogen accompanying this increase. The availability of these high-protein rices has made possible the initiation of studies on fine-grinding and air-classification to obtain very high-protein rice flours. Promising results have been obtained in early studies.

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Area 3 FORAGES AND FEED-- PROCESSING AND PRODUCTS

Problem. Fresh forage crops are the richest natural sources of a wide variety of nutrients essential to farm animals. The bulk of these crops, however, is preserved by such inefficient processes (hay making and ensiling) that 10 to 50% of the original dry weight and much larger amounts of certain valuable nutrients and growth-promoting factors are lost before the animal consumes the products. Dehydration is currently the only practical means for preserving a high percentage of the value of forage crops. Poultry and swine producers, aware of the value of dehydrated forage in feeds, nevertheless restrict the use of this product because of its high fiber and growth-inhibitor content. The livestock producer needs, and therefore the forage dehydrator needs to produce, feed ingredients from forages, tailored to specific classes of farm animals. Intensive basic and applied utilization research are needed to develop new methods for processing forages to produce: (1) high-value, fiber-free juice or low-fiber products for non-ruminant animals; (2) low-cost products, rich in fiber which has been treated to make it highly digestible, for ruminants; and (3) a growth-stimulating supplement for ruminants, taking advantage of the presence in forages of such biologically active factors as the fiber digestion factor and the growth-promoting factor which appears to be coumestrol, the newly discovered forage estrogen. Forage products for ruminant feeding would be specifically designed for the mechanized feeding operations which will be essential for the 45% increase in livestock production needed to meet the projected 1975 requirements. Development of new processes and improved forage products would stimulate the production of large tonnages of forages as cash crops on high-value land now being used for crops currently in surplus.

USDA PROGRAM

Current research in the Western Utilization Research and Development Division includes both basic and applied studies on forages, principally alfalfa and other legume forages. The research is conducted at the Division headquarters at Albany, California, under contract at Berkeley, California, and under the P.L. 480 grant program in Edinburgh, Scotland. Basic, compositional studies deal with the potent estrogen, coumestrol (discovered by Department scientists), and other phenolic compounds present in forage legumes. The value of coumestrol-rich alfalfa as a growth stimulant for ruminants is being studied, and has been aided partially by funds granted by the American Dehydrators Association. Also under study are additional biologically active forage constituents such as the chick growth-promoting factor in forage juices and alfalfa saponins which depress chick growth. The mechanism of action of forage antioxidants is being studied under

contract at Berkeley, California. Processing of forages by both "wet" (juicing) and "dry" (turbo-milling and air classification) methods is being investigated, aided in part by a grant of funds from the Department of Agriculture and Inspection, State of Nebraska.

The Federal program of research in this area totals 8.1 professional man-years. Of this number, 6.6 are assigned to chemical composition and physical properties; and 1.5 to new and improved feed products (this program is augmented by two professional employees whose salaries are provided by the Department of Agriculture and Inspection, State of Nebraska). In addition the Division sponsors, under P.L. 480, 2.0 professional man-years of research on forage composition.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 3.6 professional man-years distributed as follows: chemical composition and physical properties 1.8; new and improved feed products 0.3; and new and improved processing technology 1.5. Composition research involves the development of new or improved routine analytical methods, and determination of the amounts, roles, and physiological effects of non-nutritive additives in commercial feeds. Feed product research deals with the effects of such factors as fineness of grind upon pellet durability and physical quality. Processing studies are concerned with changes in form, pelleting, problems of segregation during mixing and conveying and development of new mixing procedures.

Industry and other organizations are estimated to be conducting research on forages with expenditures equivalent to about 6 professional man-years. One-third of this effort is expended by chemical companies in the development of antioxidants suitable for use with forages. The remainder of the industry effort is directed at the improvement of machinery for harvesting, chopping, dehydrating and pelleting alfalfa or other forages.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Coumestrol. Studies have continued aimed at determining the factors affecting the coumestrol content of alfalfa. Variables investigated included: variety, cutting, stage of growth and geographical location. The studies of samples from the 1960 crop year, in cooperation with Crops Research Division, ARS, indicated that a highly complex situation exists with respect to environmental effects on coumestrol level. For this reason the studies were extended to include samples grown in 1961. Data obtained appear to indicate

that the only effective controllable variable is the stage of maturity at which the alfalfa is cut (coumestrol increases fairly consistently in alfalfa past full bloom). More definite conclusions should be possible following a thorough statistical analysis of the data, now in progress in the Crops Research Division. The American Dehydrators Association in 1961 repeated its 1960 grant of $3000 to support analytical work required in assaying the several thousands of samples involved in this experiment. Coumestrol assays were greatly speeded by a markedly improved fluorometric assay technique; this procedure is sufficiently simple and rapid for control use in the dehydration industry.

Cooperative, large animal feeding trials were conducted for a second year, using high- and low-coumestrol dehydrated alfalfa obtained after screening production lots at commercial dehydrating plants. Scientists at Oregon State University confirmed their first year findings that high-coumestrol meal produced higher growth rates in wether lambs than did low-coumestrol alfalfa meal. Results with beef cattle at the Nebraska Experiment Station in the 1960-1961 study were negative in this respect although positive results had been obtained the two preceding years. No reasons for this inconsistency were immediately apparent, but levels fed and fiber content are suspect. In order to eliminate such factors, a large quantity (about 45 tons) of very high-coumestrol dehydrated alfalfa was obtained from cooperating commercial sources, and processed (under contract) to yield a coumestrol concentrate for use in feeding studies. The composition and biological activity of this concentrate are being determined preparatory to further cooperative large animal feeding trials.

2. Other Phenolic Compounds. In addition to coumestrol, forages contain a number of other compounds possessing "estrogenic" activity, including the isoflavones, genistein, formononetin, daidzein, and biochanin A. Although the biological activity of these compounds is considerably less than coumestrol's, their presence in alfalfa contributes to the total effect produced in a consuming animal. In certain clovers, genistein or formononetin occurs in such large amounts that they account for the major portion of the estrogenic activity. Furthermore, it was found that the biological activity (mouse assay) of a sample of high coumestrol alfalfa was greater than the sum of activities expected from the content of coumestrol plus known isoflavones. Preliminary studies have indicated the presence of about 125 other compounds in acetone extracts of forages; the isolation and characterization of these substances is now in progress, and biological evaluation studies are planned.

3. Chick Growth-Promoting Factor. Alfalfa juice fed at level of 1.5% stimulated the growth of test chicks. Dehydrated alfalfa showed some activity but it appears that dehydration has a destructive effect

on the responsible factor. Significantly, low-coumestrol alfalfa samples were as effective as those with high coumestrol levels. Acetone extracts of alfalfa, known to contain essentially all of the coumestrol and estrogenic isoflavones, proved to be inactive in promoting chick growth. Furthermore, it has been shown that the growth-promoting factor is concentrated in the acetone-insoluble fraction or in water extracts. Results of these experiments appear to offer proof that forage estrogens are not involved in chick growth stimulation, and that another factor is operative. A study of the composition of the acetone-insoluble fraction will be undertaken in an attempt to isolate the chick growth-promoting factor(s).

4. Saponins. A new paper chromatographic technique has been developed which yields a correction factor to be applied to data obtained from the gravimetric analysis for total saponins in forages. This correction was made in data on total saponin contents of some 300 samples resulting from a cooperative study (with Crops Research Division) on the relation of saponin level of alfalfa to variety, cutting, growth location, etc. Preliminary data indicate that alfalfa variety and cutting are major determinants of saponin content. Detailed conclusions should be possible following statistical analysis of the data by Crops Research personnel. Chick feeding experiments have shown that crude extracts containing the water-soluble saponins of alfalfa produce little growth inhibition. The tests will be repeated using purified preparations. Because of the insolubility of the soyasapogenol-type saponins, their bioassay has been very difficult. It has now been found that these saponins can be solubilized by the accompanying use of certain bile salts, and it is planned to determine the chick growth-inhibiting properties of these saponins. Large-scale preparation of both types of saponin for these studies is in progress. Only a very small amount of saponin was found in bird's-foot trefoil, which is in accord with the observation of livestock producers that ingestion of this legume does not cause ruminant bloat. Similarly, low or no saponin contents were shown for berseem clover, vegetative oats, reed canary, and coastal Bermuda grass.

5. Mechanism of Antioxidant Action. Basic studies of the mechanism of action of forage antioxidants have been conducted under contract at the University of California, Berkeley. Also investigated were the roles played by certain constituents, occurring naturally in forages, which might act as antioxidants or as synergists for added antioxidants. Compounds studied were: a) antioxidants: ethoxyquin, diphenylphenylenediamine (DPPD), nordihydroquairetic acid (NDGA), propyl gallate, tertiary-butyl hydroxyanisole (BHA), tertiary-butyl hydroxytoluene (BHT), alpha- and gamma-tocopherols, ubiquinones and a series of substituted bis-phenols; b) synergists: oleic, palmitic, citric, sorbic and ascorbic acids, and octadecylamine, dioctylamine, tri-n-octylamine and tri-iso-octylamine. One of the important findings arising from

this research was that while NDGA is a better antioxidant than ethoxyquin for pure triglycerides, if as little as 1% free fatty acid is present, the reverse is true. This helps to explain the inconsistencies found when comparing antioxidants for their value in protecting different food and feed products. Since dehydrated alfalfa contains about 4.5% free fatty acids, it is now obvious why ethoxyquin is much more effective an antioxidant for this product than is NDGA. Furthermore, none of the synergists tested increased the effectiveness of ethoxyquin in preventing the oxidation of β-carotene in dehydrated alfalfa. Thus, antioxidant effects in alfalfa may be influenced by the kinds and amounts of other alfalfa constituents or their physical distribution in the various plant structures. This contract has been terminated, and additional future work on oxidation and antioxidant activity will be necessary to clarify the mechanisms involved.

6. Survey of Forage Nutrients. Comprehensive analytical data for 24 forage samples including alfalfa, several grasses and grass-legume mixtures have been published in a Department Technical Bulletin. This line of work and its covering line project have been discontinued because of loss of personnel. However, a related study, that on structural analysis of alfalfa polysaccharides, has recently begun under Professor E. L. Hirst at the University of Edinburgh, supported by Public Law 480 funds. Dr. Hirst, a world-renowned polysaccharide chemist, will be characterizing the carbohydrase enzyme systems present in alfalfa. Making use of these enzymes, as well as chemical techniques, information will be sought regarding the chemical structure of pectin-like materials, pentosans, hemicelluloses, etc., and also the manner and extent of linkage of the individual polysaccharides to one another and to lignin.

B. New and Improved Feed Products

1. Forage Juice Concentrates. Earlier studies were extended on the use of a pilot scale sugar beet diffuser for preparing water extracts from fresh chopped alfalfa. Modifications in the Silver slope diffuser improved the collection of the dilute extract, and permitted automatic control of the feed end diffusion chamber heating. Diffusion capacity is adequate for the preparation of relatively large amounts of water extracts of forages for use in bioassay and cooperative, large animal feeding experiments. Presently low concentration capacity will be remedied with the installation of turbo, swept-film evaporation equipment which has been ordered. These studies are now in abeyance pending the assembly of complete processing equipment.

2. Air-Classified Forage. Dry fractionation of dehydrated alfalfa was accomplished, in an exploratory experiment, by grinding through a quadriplex mill and screening. The sample used had an initial

protein content of 21.2%. The coarse fraction obtained contained 14.1%, while the finer material contained 23.7% protein. Crude fiber contents of the coarse and fine fractions were 37% and 19% respectively. Preliminary experiments also demonstrated that dehydrated alfalfa can be reground in the Pillsbury Model H-7 turbo grinding equipment if the starting material is finely enough ground (e.g., particles passing through a No. 24 screen but retained on a No. 50). With such material, a maximum feed rate of about 0.5 pounds per minute was obtained for this turbo grinder. Proximate chemical composition of the fractions produced indicate considerable promise for dry-fractionated forage products. Currently major effort is being directed toward determining the distribution of nutrients in hand-dissected plant parts and toward development of improved methods of separating stems and petioles from leaves. Methods are being developed for microscopic, semi-quantitative estimation of protein constituents and identification of cell fragments of finely ground dried forages to facilitate the determination of the effectiveness of various grinding and fractionating procedures to be tested in these studies. This research program is being expanded with financial support from the Department of Agriculture and Inspection, State of Nebraska.

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Area 4 WOOL AND MOHAIR -- PROCESSING AND PRODUCTS

Problem. A principal reason why synthetic fibers are making increasing inroads into many of the traditional markets for wool and mohair is that fabrics made from synthetics have certain inherent desired qualities. Some of these are shrink resistance, quickness and smoothness of drying, wrinkle resistance, and ability to hold pleats and creases. Despite the superiority of wool and mohair in tailorability, comfort in wear, appearance and hand, they are lacking in some of the requirements for ease-of-care performance. Moreover, in present processing practices and in many of their uses, wool and mohair are subjected to conditions which result in damage, distortion or weakening of the fibers, and in undesired changes in performance and appearance of fabric.

Needed are practical treatments of wool and mohair to overcome these problems; for example, modifications that give durably wrinkle-resistant lightweight wool fabrics, treated fabrics that are resistant to mussing in wear and in laundering, more resistant to soil. acids, alkalies, wear, pilling, and abrasion; fabrics that have greater resistance to felting and relaxation shrinkage; wools durably resistant to yellowing, to insects, and microorganisms. Needed also are new types of fabrics, woven and non-woven, for industrial and other uses, made from natural wools, blends of wool with modified wools, and with other fibers. Development of new and improved wool and mohair products and processing methods will require fundamental information on the chemical, physical, and structural nature of these fibers. If a stable sheep and wool industry is to be sustained, mills must be supplied with needed processing information on how to produce new and better wool products more efficiently. In addition, inroads have been made in wool markets because of uniformity of price and quality of synthetics and the detailed information which producers of synthetics supply for processing these fibers on textile machinery for wool.

USDA PROGRAM

In the Western Utilization Research and Development Division, a broad program of basic and applied research on wool and mohair is conducted at the Division headquarters at Albany, California, by contract in Dedham and Lowell, Massachusetts; Salt Lake City, Utah; and Washington, D. C.; and by grant funds under P.L. 480 in England, France, and Finland. Fundamental research is conducted on wool and mohair to relate chemical composition and structure, molecular structure, physical structure, physical properties, and surface properties of both normal

and chemically modified fibers to the performance characteristics of the fibers in yarns, knitted andwoven fabrics, and nonwoven forms such as felts. Additional fundamental research is conducted on the chemical modification of wool and mohair to impart resistance to degradation by heat, light, and chemical environments encountered in use, and to improve use properties such as washability, crease retention, wrinkle recovery, and resistance to staining, abrasion, and insect attack. Applied research is conducted to develop practical processes for the chemical or physical modification of wool and mohair fibers, yarns, fabrics, and felts; to develop processing procedures for the modified fibers; and to develop new and improved products from the modified fibers; all to increase the utilization of wool and mohair. Recent development of a practical process for making wool fabrics machine washable is typical of the applied research.

The Federal program of research in this area totals 40.6 professional man-years. Of this number, 17.7 are assigned to chemical composition and physical properties; 13.9 to new and improved textile products, and 9.0 to new and improved textile processing technology. In addition, the Division sponsors 11.5 professional man-years of research under P.L. 480 including 6.5 on basic studies and 5.0 on application of research findings.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations have not reported work on the utilization of wool and mohair in recent years.

Industry is conducting very little research on wool and mohair. With the advent of synthetic fibers, wool processors lost interest in wool per se and undertook processing of the particular fibers that were in demand. Producers of synthetics conducted the research needed to adapt wool machinery to processing of synthetic fibers and furnished the information to the industry. This service, in combination with a serious decline in the financial strength of the wool industry, resulted in a shift of scientists from wool research to quality control, mill troubleshooting, and short range developmental work. U. S. textile machinery manufacturers devote only a small proportion of their research effort to improved wool processing equipment. In summary, wool research has declined seriously over the years, and much of the research effort on wool is pointed toward improving synthetic fibers rather than toward increasing the utilization of wool and mohair. It is estimated that not more than the equivalent of 50 professional man-years are involved in wool research that will benefit the American sheep industry as compared to the equivalent of 2,000 or more professional man-years estimated as being expended on synthetic fibers competing with wool.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Effects of Light and Heat. It is well known that light discolors wool, but it is not well known that light can also bleach wool. Upon exposure to ultraviolet light of wavelength 254 millimicrons, wool first turns greenish yellow, but the green color disappears within a few hours leaving the wool yellow. With longer wavelength ultraviolet light (334 millimicrons), wool yellows without a trace of the fugitive green color. With still longer wavelength ultraviolet light (365 millimicrons), wool yellows first and then bleaches. Visible violet and blue light bleaches wool rapidly. Bleaching occurs similarly in ordinary wool or in wool extensively yellowed by heat or ultraviolet light. Temperature, time, and intensity of irradiation have a complex effect on the colors produced in the wool. Presence of ozone, weave of the fabric, or grease content of the fabric had negligible effect on the color reactions. A better understanding of these color reactions may provide guides for ways of protecting wool against discoloration in use.

Earlier work on the protection of wool from discoloration by light uncovered a number of benzophenone compounds that were effective absorbers of ultraviolet light. It has since been shown that fabrics are protected by these compounds only in amounts that are impractical and uneconomical. The project has consequently been terminated.

Correlation of color changes produced by irradiation of wool with occurrence of free radicals (unpaired electrons) has been revealed by electron paramagnetic resonance spectroscopy (EPR). Many wools have been examined by this technique and found to contain a low but stable concentration of free radicals. The natural free radical is not produced by irradiation because similar concentrations were found in wool from sheep grown in complete darkness for this work by the Animal Husbandry Research Division's Sheep and Fur Animals Research Branch. EPR confirms the finding that irradiation of wool with ultraviolet light produces complex effects. The EPR spectrum of wool irradiated at low temperature persists until the wool is warmed. The spectrum is then similar to that obtained by irradiating the wool at the higher temperature. When wool is irradiated at room temperature and then cooled, the EPR spectrum obtained at the lower temperature is similar to the spectrum measured at room temperature. If wool irradiated at low temperature is warmed and then cooled again, the EPR spectrum measured at low temperature is essentially the same as that measured at room temperature, showing that the change in spectrum with temperature is irreversible.

When wool is exposed in air to ultraviolet light for extended periods,

a substantial part of its weight cannot be accounted for by amino acids obtained by hydrolysis. All of the original amino acids except glycine and alanine are degraded by the ultraviolet irradiation. About half of the degradation products have been identified, the most prevalent being alpha-aminobutyric acid, homocysteic acid, S-methylcysteine, and 1-methylhistidine.

Under contract, an attempt was made to determine the effects of heat on wool, mohair, other proteins, amino acids, and peptides by means of differential thermal analysis. All wool and mohair samples showed only two endothermic peaks, one ascribed to the release of bound water and the other to gross structural breakdown of protein. It was not possible to distinguish among wools or between wool and mohair by the technique.

2. Chemical and Molecular Properties. Basic research on the wool molecule is underway, both at the Western Utilization Research and Development Division, and abroad under P.L. 480 grants. At Albany wool proteins are being fractionated and stabilized, the molecular weights and amino acid compositions of the various fractions are being determined. Results related to molecular weight are being calculated by the Computing Center, Agricultural Research Center, Beltsville, Maryland. In France, a study is in progress to determine the sequence of amino acid building blocks in proteins of wools selectively degraded under controlled acid conditions. The purpose is to determine how chemical structure is related to quality differences among wools. Much of the work to date has been concerned with separation of constituents of the wool fiber with minimum alteration of the chemical structure. In England, a study is in progress to establish the role that sulfur plays in the unique characteristics of wool. From 10 to 15% of the sulfur content of wool cannot be accounted for by ordinary analysis. The investigators have been able to isolate a partial oxidation product containing sulfur from wool hydrolysates but have been unable to identify the product because of its lability. Other research in England involves measurement of self-diffusion of ions of various sizes into keratin proteins under conditions that reveals protein structural differences. This work is yielding new information and may ultimately lead to improved methods for chemical treatment, chemical modification, and dyeing of wools.

The technique of proton magnetic resonance spectroscopy (PMR) yields information on the location of protons or hydrogen nuclei in molecules. The relatively high capacity of wool to bind moisture is one of its important characteristics, but the mechanism of moisture binding is poorly understood. PMR is yielding information on the location of water in wool. For example, with wool containing 10% moisture, the water seems to be bound in clusters to the wool structure and to undergo surface migration. Additional studies are in

progress on PMR spectra of relaxed and stretched dry wools and on amino acid constituents of wool, particularly glycine and glycines deuterated in various positions. The latter will help in the interpretation of the complex PMR spectra of wool. This work may lead to an understanding of the variation of mechanical properties of wool with moisture content.

3. Measurement of Fiber Fineness. Wool fiber fineness and fineness distribution, an essential part of processing research, have routinely been determined by standard microscopic methods followed by calculations requiring approximately 20 minutes. This time has been reduced to 2 minutes by development of a special type graph paper to calculate results. A more precise method which utilizes a Coulter Counter, an electronic device for measuring particle size and size distribution, has been adapted to determination of fiber fineness and fineness distribution. Operating conditions have been established and the reproducibility and error in the instrument have been defined. Analysis of the data shows that the instrument provides accuracy and precision equivalent to that of the best operators using the standard microscopic method, without the inherent subjective error in the standard method. The standard microscopic method requires two to three times longer than the Coulter Counter to measure the fineness and fineness distribution of the fibers in a sample of wool.

4. Mechanical Damage to Wool. The way in which abrasion and wear break down wool structure is poorly understood. Electron paramagnetic resonance spectroscopy (EPR) is yielding information that helps to explain wear and abrasion. When wool is abraded, the concentration of free radicals is increased as is the case when wool is exposed to light or heat. If wool is severely damaged, i.e., ground to a powder, the EPR signals are similar to those obtained when wool is exposed to ultraviolet light of short wave length or to X-rays. Conventional processing operations such as carding or drawing increase the intensity of the EPR signal. Both the irradiation and mechanically-induced free-radical signals disappear if the wool is annealed at 100° C. for several hours, allowed to absorb substantial amounts of water vapor, or wetted out in water. The free radicals arising from irradiation or mechanical damage are believed to be associated with the amino acids, tyrosine and cystine.

5. Physical and Mechanical Properties. Measurements have been made of the frictional properties of wool fibers after application of the WURLAN or IFP polyamide treatment. (See paragraphs B-1 and C-1 of this area report for treatment details.) It was found that the surface friction of the treated fibers was much higher than

that of untreated fibers, but the differential friction (with and against the scales of the fiber) was the same for treated fibers as for untreated fibers. In the past, shrink resistance in a wool fabric has been attributed to reduction of differential friction by various means. Present results show that shrink resistance can be obtained without changing the differential friction. A simple treatment of felting shrinkage as affected by surface friction has been useful in describing the rate of felting of loose top into a ball. (See paragraph C-2 of this area report.) A similar interpretation provided semi-quantitative results when applied to fabric shrinkage, but the effect of agitation introduced a complexity that has not yet been resolved.

Research is in progress on the dynamic mechanical properties of keratins toward defining the interrelations among temperature, moisture content, and frequency. Measurements of the rate of damping of a fiber-supported torsion pendulum show two regions of interest. One, at about -100° C., is due to water bound strongly to the keratin lattice, contributing to a 10% decrease in modulus of elasticity. The other, occurring from 50° to 200° C., depends upon moisture content, and involves a decrease in modulus of elasticity as much as 90%. This is related to loosely-bound water and reflects the great effect of water on mechanical properties.

B. New and Improved Textile Products

1. Shrink Resistant Fabrics. Research has continued on a technique known as interfacial polymerization (IFP) for applying polymers to wool top, yarns, and fabrics. The primary purpose is to make wool fabrics that are fully machine washable, although muss resistance, improved abrasion resistance, and other improvements can be obtained from the treatment. One of the first polymers tested, poly(hexamethylene sebacamide), has given results as good or better than more than 20 other polyamides that have been evaluated. The IFP technique has also been used for applying a wide range of different polymers to wool fabrics, including polyurethanes, polyureas, polyesters, polycarbonates, and copolymers. Several of the polyurethanes have, in preliminary tests, shown results comparable to those obtained with poly(hexamethylene sebacamide). This research will continue because polyurethanes are potentially cheaper than poly(hexamethylene sebacamide). Evidence is accumulating that the IFP polymers are grafted to the wool, a factor contributing to the durability of the treatment. By addition of appropriate chemicals to the solutions used for the IFP treatment, it has been possible to impart shrink resistance, mothproofness, and water repellency to wool fabrics in a single treatment. Tests show that fabrics stock dyed with chrome and neutral premetallized dyes are usually resis-

tant to bleeding and shade change during IFP treatment. Milling dyes should be applied after the IFP treatment. In most cases fabrics can be dyed satisfactorily after application of poly(hexamethylene sebacamide). The IFP treatment ordinarily has little effect on the washfastness or lightfastness of dyes. The process for applying the IFP to fabrics is described in paragraph C - 1 of this area report.

The epoxy-polyamide treatment for shrink resistance has been shown to be less suitable for most fabrics than the IFP polyamide treatment. In consequence, research on the epoxy-polyamide treatment is being limited to fabrics such as blankets where the treatment can be applied in conventional equipment such as dye becks in plants where padders for the IFP treatment are frequently not available. Part of the research on the epoxy-polyamide treatment was done under contract by the Fabric Research Laboratories, Inc., Dedham, Massachusetts. Results included comparison of padding and exhaustion as methods of applying the resin, effect of fabric construction on shrink resistance and handle of the treated fabric, and analysis of causes of stiffness in treated fabrics.

2. Chemical Modification of Wool. Research is continuing on chemical modifications of wool to impart new properties. For example, greater resistance to acids and alkalies could lead to new industrial uses for wool. Both dimethyl sulfoxide and dimethylformamide are good solvents for reacting wools with a variety of acylating reagents. Wool reacted with isocyanates has shown increased resistance to acids, alkalies, and hypochlorite, dye resistance, and, in some cases, shrink resistance. In a series of n-alkyl isocyanante-modified wools, it was observed that the alkali solubility of the treated wool decreased with increasing alkyl chain length in the isocyanate. In general, diisocyanates impart more chemical resistance to wool than monoisocyanates. Isocyanates will react with wool in gamma-butyrolactone but at slightly slower rates than in the two solvents mentioned above. Wool has also been reacted with a variety of organic acid anhydrides. Wool thus modified is more resistant to acid and less resistant to alkali than untreated wool. At high uptakes, the alkenylsuccinic anhydrides impart moderate shrink resistance to wool. Wool has also been modified with lauroyl, myristoyl, and stearoyl chlorides, and with adipoyl and sebacoyl dichlorides. Fabrics so treated show decreased resistance to hot alkali, increased resistance to hot acid, and shrink resistance at high uptakes as compared to untreated wool. Although some of the chemical modifications outlined above improve certain properties of wool, none appears to have commercial potential. Problems include the need for hot, anhydrous, organic solvents, polymeric products from side reactions, and loss of strength and resilience in the wool.

3. Durable Creases in Wool Fabrics. As part of a program to improve wool's easy-care properties, processes have been investigated for imparting durable creases to wool fabrics. Although a wide variety of chemicals have been screened, including cross-linking agents for reduced wool, ethanolamine in dilute aqueous solution performs as well or better than other agents tested. No fully satisfactory treatment is now available for durable creasing of wool. Ethanolamine sulfite, independently recommended by the Wool Bureau, has little effect on fabric color, but the treated fabric develops an odor upon wetting. Ethanolamine does not cause an odor problem, but tends to yellow the fabric slightly and to make a slightly harsh crease. Nevertheless, the Quartermaster Corps had forty pair of uniform trousers durably creased with ethanolamine under our supervision for test. We are informed that the QM is writing specifications for the durable creasing of army trousers with ethanolamine. Development work was also carried out on objective measurement of creases and wrinkles in fabrics. An instrument was constructed that traces the fabric contour and computes any desired factor to describe the crease or wrinkle. Crease shape can be described easily, but no definition of wrinkledness has yet been found that correlates fully with the subjective judgment of a test panel.

4. Treatment of Wool Top for Shrink Resistance. As outlined in paragraph C-1 of this area report, a practical process has been developed for applying the WURLAN or IFP polyamide treatment to woven fabrics and to knitted yardage. The treatment is also effective on knit piece goods such as socks and sweater bodies. However, no practical way has been found for applying the treatment to piece goods. As an alternative, research was initiated on applying the treatment to wool top, using equipment similar to that used for backwashing of top. Considerable success has been obtained, although more developmental study is needed to make the treatment practical under commercial conditions. The treated top spins satisfactorily and the treatment does not seem to affect yarn uniformity. Fabrics knitted from yarns made from treated top show good resistance to felting shrinkage. Since the polyamide film increases the surface friction on the wool fibers, yarns from treated top require less twist for equal strength than yarns from untreated top. This could lead to reduced costs since the production rate of a spinning frame is related to the amount of twist in the yarn being spun. High-twist yarns from treated top can be woven into hard-finished worsteds that show unusual and desirable crispness and handle. Application of the WURLAN treatment to top profoundly affects the fulling properties of subsequent fabrics. Fabrics can be made that show good shrink resistance even though loosely constructed, and fabrics can also be made that cannot be fulled. This work will lead directly to entirely new types of fabrics from wool.

5. Improved Carbonizing of Wool. Under contract with the Harris Research Laboratories, Inc., Washington, D. C., a project was completed on improved carbonizing of vegetable matter in wool. It was found that there are three main factors in the sulfuric acid carbonizing process that govern the degree of strength loss of the wool: (1) the acid content of the wool entering the dryer; (2) the moisture content of the acid-containing wool as the material enters the dryer; and (3) the air temperature used to dry the wet, acid-containing wool. By proper adjustment of these conditions, it was found that burrs could be completely carbonized without causing any measurable strength loss of the wool. It was also shown that the rate of diffusion of sulfuric acid into wool was significantly slower than the rate of sorption of acid onto the burrs, especially when the acid solution temperature is between 50° F. and room temperature. Advantage was taken of this difference in rate of sorption to deposit the required amount of acid on the burrs while a relatively small amount was taken up by the wool. Thus, a new and faster treating procedure, differing from conventional commercial practice in several aspects, was suggested: 7 to 7.5% sulfuric acid in the treating solution, at 50° F., with immersion of the burry wool for 15 to 60 seconds instead of 3 to 7 minutes or more. A further contribution of this research is a clarification of the role of wetting agents in the acid bath. Wetting agents per se do not protect the tensile strength of the wool, but their beneficial effect is a result of their reducing the water content of the extracted, acid-treated wool prior to drying and baking. A number of other factors in processing also were clarified. For example, it was shown that all of the wool damage (loss of strength), if any, occurred during the drying phase of the carbonizing process. There was no further loss in strength after baking provided that the wool was dried to a moisture content below 14% before it was baked. Drying time was not an important factor, but drying temperature was of great importance especially when the wool contained more than 5% acid. The optimum drying temperature was between 130° and 150° F. The optimum baking time and temperature for the dried wool was three minutes at 300° F., when the air velocity in the oven was 400 linear feet/minute and the loose wool was in a layer 1" to 1-1/2" in thickness, and when the acid content of the burr was between 2.5 and 3.0%. As might be expected, fine wools were more easily damaged by carbonizing than were medium or coarse wools. Thus, the fine wools (64 grade and finer) require somewhat milder processing conditions (lower acid content or lower drying temperature) than coarser wools. The contractor's final report presents recommendations for processing conditions applicable to pilot plant and mill trials.

6. Bleaching of Wool. A research contract was initiated at the Lowell Technological Institute, Lowell, Massachusetts, on improved

procedures for bleaching wool. A supply of discolored wool was obtained and portions were processed into top, yarn, and fabric to be used in the project. Exploratory tests have been made on the bleaching of top in which time, temperature, pH, and peroxide concentration were the variables. Measurements were made of the reflectance and alkali solubility of the treated wool, and the consumption of peroxide. The project has not yet reached the stage where broad conclusions can be drawn.

7. Improved Finishing Treatments for Wool Fabrics. Supported by a P.L. 480 research grant in Finland, a study is in progress to determine the influence of different finishing procedures on wool fabric properties, and to develop a finishing procedure that imparts to the finished fabric optimum properties with regard to appearance, handle, and tailoring and weaving qualities. The experimental fabrics for the project are being designed and woven and preliminary dyeing studies are in progress. No conclusions have yet been reached.

C. New and Improved Textile Processing Technology

1. Treatment of Fabrics for Shrink Resistance. A practical process has been developed for applying the IFP polyamide treatment to wool fabrics for control of felting shrinkage. (See paragraph B-1 of this area report for additional discussion of the IFP treatment.) Through statistically-designed experiments, important process variables were identified and evaluated. The treatment is best applied with two padders in tandem. In the first padding step, the aqueous solution of hexamethylene diamine should be heated to the range of 100 to 130° F., and the fabric should be skyed to provide a time lapse of 20 to 30 seconds between immersion and padding of the fabric. The load on the padder rolls should be adjusted to give 50% or less wet pickup of solution by the fabric. Neither solution temperature nor time lapse is important in applying the sebacoyl chloride solution in the second padding step. Under the process conditions specified, fabric has been continuously and satisfactorily treated at rates as high as 25 yards per minute.

The IFP treatment was announced in a new release of October 12, 1960, that brought in well over 100 inquiries. About 150 different fabric samples were treated for manufacturers who expressed interest in the process. Demonstrations of the treatment were held in the Wool Processing Laboratory at Albany, California, and Department scientists supervised mill trials in plants of several major textile producers A technical conference featuring the IFP process was held at Albany on February 9 and 10, 1961. About 75 people were in attendance. The IFP polyamide treatment has since been designated as the WURLAN process.

2. Felting of Wool. Typical woven wool fabrics and nonwoven felts could not be made if wool did not have the ability to felt. However, this characteristic is responsible for the continuing shrinking of wool fabrics during washing. Knowledge of how to promote as well as inhibit felting is obviously important in the development of new and improved wool products. Study of the fulling process has shown that temperature is the most important factor affecting rate of fulling. Periodic interruption of the fulling operation and trap weight had little effect on fulling rate. It was clearly shown that the type of finish produced by fulling depends upon both rate and extent of fulling. Work was continued on a rapid method for measuring the feltability of various wools. The method involves suspension of a weighed amount of wool in a bottle containing the test solution and shaking of the sample for definite periods of time. The wools form balls whose diameters are related to felting ability. Although results show that the method is useful, none of a variety of compounds tested in the felting solutions were found to have a pronounced effect on felting rate. Artificial removal of crimp from wool increased feltability appreciably. The IFP polyamide treatment of wool fibers decreased their feltability markedly.

3. Fabric Construction. Both yarn and fabric construction greatly affect the performance of a fabric as well as the response of the fabric to chemical treatments used to impart easy-care properties. During screening of fabrics from various processors to determine the amount of polyamide required to control shrinkage, all-wool fabrics were found that exhibited from 5 to 75% area shrinkage in our standard wash test. The amount of polyamide required to control shrinkage in different fabrics has ranged from 0.25 to 2.5%, the lower amounts being adequate for some blend woolens and the higher amount being required for hard-twist, tightly-constructed worsteds. It is anomalous that fabrics showing comparatively little shrinkage without treatment require substantial amounts of polyamide for further control of shrinkage. This shows that a truly washable wool fabric should be designed for this specific end use, starting with the construction of the yarn and continuing through construction of the fabric. Similar conclusions have been reached by processors working with other shrink-resist treatments. The WURLAN treatment, however, has controlled shrinkage in a much wider range of fabric constructions than any other shrink-resist treatment.

Further information was obtained on relations among fabric properties and the amount of epoxy-polyamide resin needed to control shrinkage. The study encompassed 19 different fabrics, including woolens and worsted, tight and loose constructions, light and heavy fabric weights, and dyed and undyed samples. It was shown that the amount of epoxy-polyamide resin needed to control shrinkage

is an inverse function of the product of fiber diameter, ends per inch plus picks per inch, and the square root of yarn tex. The coefficient of the equation is dependent upon the dye on the fabric.

4. Uniformity, Strength, and Nature of Yarns. Uniform, strong yarns are necessary for making worsted fabrics, particularly lightweight fabrics. Studies have been made to determine the relations among fiber properties and the uniformity and strength of yarns produced therefrom. A series of yarns was made from fine wool in which twist was the variable. All yarns were of the same count. Twist was varied from the minimum at which the yarn could be spun to the maximum obtainable from the spinning frame (23 turns per inch). The top from which the yarns were made was fully characterized and the final yarns were tested for evenness, strength, and elongation. Yarns of the same count and twist were also made from fine, medium, and coarse grades of wool and were subjected to the same tests. All yarns proved to be of high commercial grade. Yarns from the fine wools showed slightly greater elongation than yarns from the coarser wools, but all yarns were similar in evenness and strength. Fabrics knitted from these yarns showed obvious differences in hand that were not reflected in any of the physical tests.

Two lots of quarter-blood wool, one from Beltsville, Maryland, the other from California, showed distinctly different processing characteristics which were reflected in the uniformity and strength of the yarns spun from the wools. By conventional processing, the Beltsville wool yielded satisfactory yarns, though not outstanding in uniformity and strength. The California wool could not be spun after identical processing because of end breakage. However, when the last pin drafting was omitted in top preparation and a double roving operation performed, the California wool produced more uniform yarns than those made from the Beltsville wool by conventional processing. Our physical tests have failed to explain the observation on processability. The two wools are of similar fineness, crimp, drafting force, and feltability, and differ only 0.4-inch in average fiber length, the California wool being the longer.

A study of the effect of lubricants and waxes on wool yarns is in progress in England with funds made available under P.L. 480. The objective is to relate knitting behavior to lubrication of wool yarns to provide a basis for the development of knitting yarns with improved characteristics and fabrics of improved appearance. To date, work under the grant deals primarily with methodology

5. New Uses for Coarse Wools. The bulk of domestic wools are of coarse grade and are not ordinarily used in suiting fabrics. New

types of yarns from coarse wools or ways of using coarse and fine wools in blends can provide new outlets for domestic wools. Attempts are being made to soften wools by chemical modification. Preliminary efforts are encouraging although few conclusions can yet be drawn. Work is also in progress on the blending of WURLAN-treated and untreated wool tops of different grades. New types of yarns and fabrics are anticipated because of the mixture of felting and non-felting fibers. Conclusions in this area of research are not yet available.

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Area 5 CITRUS AND SUBTROPICAL FRUITS-- PROCESSING AND PRODUCTS--WESTERN LABORATORY

Problem. The economic stability of the citrus and subtropical fruit industries in the Western Region is dependent upon effective utilization of fruit that cannot be accommodated on the fresh fruit market. The utilization of surplus or wholesome but blemished fruit provides the margin necessary to assure adequate returns to the farmer and continued development of stable markets. Ineffective utilization of products and continuously increasing processing costs are resulting in decreased returns to the growers. The California-Arizona grapefruit industry is encountering difficulty in disposing of both fresh fruit and processed grapefruit products. The pineapple and subtropical fruit industry in Hawaii must find practical methods for processing its products for export in order to prevent the accumulation of burdensome surpluses. The navel orange industry in California is hampered by the unavailability of satisfactory processes for the utilization of navel oranges. Juice extracted from early fruit, and during some seasons from essentially all of the navel oranges, contains unknown substances that impart an intolerable bitter flavor to juice products after mild heat-processing or after standing at ambient temperature for a short time. Large new plantings of navel oranges may be expected to aggravate the utilization problem. Deterioration of the flavor and color of these and other processed citrus and subtropical fruit products imposes severe limitations upon the economic stability of the industry.

Information is needed on the chemical composition of citrus and subtropical fruits and their products and byproducts as a basis for the development or application of new and improved methods of processing; and for the production of new and improved food and industrial products and pharmaceuticals. Special attention needs to be given to the nature of the chemical changes involved during pre-treatment, processing and handling which lead to the formation of off-flavors, -colors, and -odors in processed products.

USDA PROGRAM

In the Western Utilization Research and Development Division, a concentrated program of fundamental research on citrus and subtropical fruit and its application to industry problems is conducted at the Division headquarters at Albany, California and at the Fruit and Vegetable Chemistry Laboratory in Pasadena, California. Investigations are conducted on the composition of citrus essential oils,

flavonoid compounds and other citrus constituents that are related to off-flavors and darkening of citrus products, the natural flavor components of oranges, enzyme systems that are involved in the appearance and disappearance of constituents and structures of plant tissues, constituents of dates that affect the quality and stability of products, and the application of findings of such research to the development of new and improved citrus and subtropical fruit products.

The Federal program of research in this area totals 17.7 professional man-years. Of this number, 15.7 are assigned to chemical composition and physical properties (including one employee whose salary is provided by the Date Administrative Committee and two employees whose salaries are provided by a trade association, The Lemon Products Technical Committee), and 2.0 to new and improved food products.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations, in 1961, reported 20.3 professional man-years divided among subheadings as follows: Chemical composition and physical properties 13.7, new and improved food products 2.0, new and improved processing technology 3.6, and new and improved industrial products and feeds from byproducts of citrus and subtropical fruit processing (including waste disposal) 1.0. Basic studies are concerned with enzymes and enzyme substrates of citrus and subtropical fruits and reactions that influence the character of food products; with flavor constituents of oranges, and with the chemistry of processed olives. A lesser effort is expended on development of new and improved products and processing technologies including stabilized frozen concentrates, flavor recoveries, bacteriology of products, and recovery of valuable materials from processing wastes.

Industry and other organizations, including citrus and subtropical fruit processors, beverage and beverage-base manufacturers, equipment and container manufacturers, and flavoring manufacturers, conduct research and occasionally contract with private research institutions for special projects. They are entitled to exclusive use of data obtained or products or processes developed. Projects are also developed cooperatively with federal or other non-private research groups, in which case the results become public property. Much research of industry is concerned with applications of fundamental research findings of public institutions, or with answering

specific problems of industry, such as developing suitable containers, improving product formulations and introducing new formulations to the market. Estimated annual expenditures in this area are equivalent to approximately 130 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Citrus Essential Oils. The chemical composition and physical properties of citrus essential oils determine the quality and stability of food products made from them. Fundamental studies of citrus oils are continuing with support of the Lemon Products Technical Committee, which provides the salaries of two scientists assigned to the project. The major cause of instability of lemon oil was demonstrated to be the oxidation of gamma-terpinene to para-cymene, disproving the widely held hypothesis that para-cymene is a breakdown product of citral. This finding not only explained the failure of previous attempts to stabilize lemon oil against the development of "cymey" off-odor, but suggested new approaches to stabilize products. In the laboratory, gamma-terpinene was removed from lemon oil for an evaluation of stability by a commerical user. The modified product was reported to be 2 to 3 times more stable than an untreated control sample. Stabilizing lemon oil would make it useful in many products that cannot carry ingredients with rapid deterioration rates. Studies have also included analyses of monoterpene hydrocarbons and identification of esters and alcohols of citrus oils. Data obtained are useful in determining authenticity of lemon oils in commercial channels of trade, and may help identify source of oils. In addition, the data provide knowledge on the importance of these compounds to the flavor of citrus oils and their products. It has been demonstrated that alcohols may be artificially formed from monoterpene hydrocarbons during certain stages of processing such as in cases where the lemon oil comes in contact with the highly acid lemon juice. Thus, important contributors to flavor could be lost or reduced in intensity. It should be possible, now, to develop processing methods to reduce the conversion.

2. Citrus Fruit Composition. The color stability and flavor of citrus products are related to the flavonoid compounds found in juice. Some flavonoids, such as those in grapefruit and Seville oranges, are extremely bitter and impair the marketability of citrus products. A chemical change in flavonoids can cause a dark discoloration in processed juice products. A glucoside, phlorin, which had never before been reported as a natural constituent in plants, was isolated from oranges and grapefruit and

shown to be present in lemons. This compound is important because it turns brown in the presence of amino acids under certain pH conditions, and could be responsible for product deterioration.

New basic information has been obtained on the compounds that are most closely related to bitterness in citrus products. Certain of these intensely bitter compounds (flavonoids) can be made to undergo a chemical change that converts them to very sweet compounds (dihydrochalcones). So sweet are these compounds that they have potential commercial significance as low-calorie food sweeteners, and preliminary pharmacological tests have been run, which show these chemically-altered citrus compounds to be relatively non-toxic. Other related flavonoids are not bitter and do not become sweet by this type chemical alteration. Thus, naringin and neohesperidin are very bitter and form dihydrochalcones that are very sweet; while hesperidin, eriocitrin, naringenin rutinoside, and isosakuranetin rutinoside are tasteless and form dihydrochalcones that are also tasteless. The difference in chemical structure relating to these bitter and sweet flavors was elucidated. In the complicated molecular structure of these compounds, the characteristics of the chemical linkage between two sugars (rhamnose and glucose), determines whether or not the flavonoid will be bitter and the dihydrochalcone will be sweet.

Another bitter principle of commercial significance to the citrus industry is that which develops in Navel orange products on standing. This bitterness is related to limonoid compounds, which are commonly found also in citrus seeds, and which are now being investigated. Laboratory procedures have been adapted to this study and preliminary identification of specific compounds in Navel oranges has been made. Knowledge of such compounds is required in order to understand the nature of bitterness development in orange products and to seek means for its prevention.

3. Fruit Flavor Components. Of great importance to flavor chemistry in recent years has been the application of gas-liquid chromatography techniques. High instrument sensitivity was achieved by use of dual columns with dual-flame ionization detectors and programmed temperature control, so that it is now possible to separate and make tentative identifications of important volatile components from the headspace atmosphere of a small flask containing only a gram or two of the food product under investigation. The procedure, developed at Albany, California is sufficiently distinctive to warrant a special name. The instrumental record developed by the new procedure is called an "aromagram." Earlier extraction and concentration procedures are not required so that possibility of introducing artifacts is minimized. Sample size required is very small and food samples can be withdrawn from

processing lines at different stages or from packages during storage experience and analyzed for compositional changes that may be correlated with subjective evaluations of products. The aromagram procedure is more sensitive by a factor of ten than any method previously used. However, while this is below the sensory threshold of many flavor components, it is not sensitive enough to detect some odorous compounds at levels detectable by the human nose. Only a beginning has been made in using the aromagram in fundamental studies of fruit flavor. The complicated mixture of volatile components from oranges is being identified. In order to evaluate the flavor contribution of specific compounds, isolated substances from orange flavor essence are added individually to an orange powder which was prepared so as to prevent development of off-flavors. Evaluations are made by organoleptic, test panel methods. This work is closely connected with efforts to develop a new orange powder of superior flavor (see paragraph B, 3).

4. Composition of Dates. Darkening of dates, as they proceed through trade channels, damages their salability and reduces markets. Investigations are supported in part by the Date Administrative Committee (operating under a Federal Marketing Order) which supplies the salary of a scientist assigned to this work. Earlier work on enzymatic conversion of date sugar in order to improve product texture has been reduced to commercial practice. Current investigations are revealing the chemical nature of some of the components of dates that are known to brown under certain conditions. Three general chemical systems of discoloration have been revealed. These are enzymatic browning, indicated to be of limited duration in stored fruit, oxidative browning, and nonoxidative browning. Both of the latter two deteriorations are of long-term storage importance because of the relatively high concentration of reacting compounds. A better understanding of these reactions must be obtained in order to seek a rational control of date discoloration.

5. Texture of Fruits. Little is known of the enzymes involved in the formation of cell wall polysaccharides of plant tissue. This, in part, is due to the dearth of knowledge of the cell wall constituents themselves. Work in this complex field has been initiated, using an enzyme preparation obtained from germinating mung beans. By use of radioactive tracer techniques, the enzyme preparation was found capable of forming two polysaccharides from glucose and fructose. One of the polysaccharides was soluble in hot water, the other not. The former is apparently formed by a previously unrecognized enzymic pathway with the glucose

incorporated into the polysaccharide, intact. This polysaccharide is unusually resistant to acid hydrolysis. This study has further offered a clear insight into the nature of an alternative pathway of glucose metabolism bringing a new enlightenment to the way higher plants utilize carbon sources for cell wall synthesis.

6. Ascorbic Acid Biosynthesis in Plants. L-ascorbic acid (Vitamin C) occurs in all higher plants but little is known of its biosynthetic origin, its function, or the nature of its accumulation in fruits and vegetables. The enzymes involved in these processes are being studied by radiotracer techniques in which precursors of ascorbic acid are labeled with C^{14} and fed to living plant tissue. Ascorbic acid, later isolated from the plant, demonstrates by the amount and pattern of its radioactivity the pathways by which the vitamin is formed. It has been proven that glucose is converted, in strawberries, to ascorbic acid and not to an isomer, D-arabo-ascorbic acid as suggested by other researches. The net result of this has been to demonstrate that plants possess the astonishing capacity to convert a D-sugar to L-ascorbic acid by changing the configuration about a single carbon atom. Interrelationships are being elucidated among glucose, galacturonic acid (the main building block of pectins and important in textural quality of fruits), methyl ester of galacturonic acid (an important compound in the textural quality of fruit products), and ascorbic acid. The methyl ester of galacturonic acid is a better precursor of ascorbic acid than is the acid itself. On the other hand, both the acid and its ester function in pectin synthesis and both undergo reduction to the same compound, galactonic acid.

7. Enzymes Involved in the Ripening of Fruits with Ethylene. The effects of ethylene gas on maturation rates of harvested fruits in storage have been known for some time. The knowledge has served as a basis for controlled ripening of lemons and bananas, and accumulation of ethylene has an adverse effect on fruits to be held for long periods in cold stores. Basic research is under way to learn the biochemical nature of the ethylene effect on maturation of fruits so that its function can be better controlled and made more useful. The avocado was selected as an example fruit to study because it is available the year around, is a good producer of ethylene, and has a strong response to ethylene treatment. Avocados are exposed during maturation to ethylene synthesized with radioactive carbon or hydrogen. The labeled ethylene reacts in the maturing fruit and analyses are made to determine the reaction products. When avocados were ripened in the presence of hydrogen-labeled ethylene, 15% of the label was found in the hydrocarbon, toluene. The establishment of this hydrocarbon as a major reaction product indicates an unusual, perhaps unique, metabolic pathway involved in maturation.

B. New and Improved Food Products

1. Citrus Essential Oils. Compositional studies of citrus essential oils, supported in part by the industry (see paragraph A, 1), are applied to problems of stability of products and of establishing the authenticity of oils in trade channels. Cooperative studies are conducted as a bridge to such extension activities between basic research and commercial practice. Techniques to produce a lemon oil without gamma-terpinene were developed and led to the commercial production of a stabilized oil using another, privately-developed method. Ratios of concentration of certain natural carbonyls in lemon oil have been found to be sufficiently characteristic to be useful in identifying spurious products offered for sale as lemon oil. A deterioration of flavor-contributing terpinenes to alcohols has been observed under conditions of lemon oil recovery from peel or emulsions pressed from peel. Means for avoiding this quality loss during processing are under investigation.

2. Improved Date Products. Dates of softer texture and improved flavor can be produced from inferior quality dates by treatment under conditions that are optimum for the action of the enzyme invertase. Sucrose is inverted to fruit sugar and glucose under these conditions. This process was developed in the laboratory, supported in part by the industry (see paragraph A, 4), and has now been extended to commercial practice. Approximately 1,000,000 pounds of dates are so treated annually. If the labor-saving practice of harvesting all dates from a tree at one time is greatly extended, the portion of the crop needing this inversion treatment to improve quality should increase materially.

3. Foam-mat Drying. Over 50 agricultural commodities have been successfully dehydrated by the foam-mat drying process invented by Department engineers at Albany, California. These include orange, grapefruit, and lemon juices. Continuous, automatic equipment has been designed and constructed for continuing experimental studies. A commercial drier of similar design has been installed in a food processing plant in California and several other industry applications of this novel dehydration method are being investigated with pilot operations to develop commercial-scale equipment. Most products tested can be dried at atmospheric pressures without off-flavor development or discoloration. There is a loss of volatile flavor components. A major problem in the technology of each product so dried is to find means for adding back or redeveloping

flavor. Solid carriers, such as sugar and other carbohydrates are used to "lock in" volatile flavor components and equipment has been designed to mix and form the carbohydrate-flavor mixture. This equipment operates continuously and has advantages over previous methods by minimizing heat requirements, thereby minimizing chemical alteration of the volatile flavor components. Improved foam stabilizers, required for some products in foam-mat drying have been selected or developed to improve product quality. Drying cycles have been determined for specific products to prevent serious quality loss during processing and to increase heat and moisture transfer and mechanical efficiencies so that processing costs may be reduced. Cooperation has been given to the Winter Haven, Florida field station of the Southern Utilization Research and Development Division in applying the foam-mat drying process to orange juice.

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Area 6 DECIDUOUS FRUIT AND TREE NUTS--PROCESSING AND PRODUCTS--WESTERN LABORATORY

Problem. Fruits and nuts are valued for their unique flavor and color, and natural vitamin content. In the period of abundance at harvest time, markets are glutted and growers often do not get an adequate return for the effort and cost of production. Crops are perishable and processing to preserve their unique qualities is difficult. No processed fruit retains completely the fresh values although many highly acceptable products exist and about half of the fruits and nuts marketed in the United States are processed. This literally makes these commodities available to consumers the year around, and has opened new markets for producers. The proportion of processed commodities is steadily increasing but is dependent upon a continuing flow of new knowledge. Processing to preserve color, flavor, and texture of the raw material presents many problems generally, and each new product requires the application of much scientific and technological skill.

The freezing process for preserving certain fruits provides an excellent means for keeping the products at near fresh fruit condition. In spite of the gains in quality realized in freezing, many unsolved problems remain. The enzymatic browning of frozen peaches and the sloppy texture of frozen strawberries on thawing are two good examples.

Frozen fruits require expensive low-temperature storage and transportation facilities. The expense is greatly reduced by removing a portion of the water from the products. Examples of products developed on this principle are orange and other fruit juice concentrates which are well established in U.S. markets, and dehydrofrozen apple slices (rapid drying to 50% bulk weight and then freezing) which are just becoming well established. Many other fruits and fruit juices should be amenable to this type of processing and work on this is needed. Products of this type, however, are not so well adapted for export as those which do not require refrigeration.

The maximum weight reduction can be achieved through dehydration. The drying of fruit juices has been successfully accomplished by the vacuum puff drying and foam-mat drying processes. The latter process is under intensive study, because it can be carried out at atmospheric pressure and consequently offers a potential economy in processing. This process must be worked out in detail for many, as yet untried, fruit purees and juices and on pilot-plant scale for those products that show promise. Flavor recovery and the incorporation of recovered flavor in solid carriers for addition to the dried products are problems requiring technological and basic chemical

study. Essence recovery techniques developed for fruit juice concentrates are not completely satisfactory for this purpose.

Dried fruits and canned fruits are currently widely used in the U.S. and abroad. The popularity of dried fruits overseas and in this country would be enhanced if stable, higher moisture dried fruits were available and if lower levels of sulfur dioxide could be used without loss of quality. Control of mold spoilage in high-moisture dried fruit requires effective antimycotic agents.

Container costs for canned fruits limit considerably the shipment of these products overseas. A solution of the container problem is needed and may be found in the use of lightweight fiber, foil, or plastic containers and aseptic filling procedures.

A pressing problem of fruit growers is the need for new varieties of tree fruits and berries suited to processing and resistant to diseases endemic to each region of production. Utilization research is required in cooperation with farm research to assure growers of a market for fruit in the processing industry.

USDA PROGRAM

In the Western Utilization Research and Development Division, a broad program of basic and applied research on deciduous fruits and tree nuts is conducted at the Division headquarters at Albany, California, in field stations at Pasadena, California, Prosser and Puyallup, Washington, by contract in Honolulu, Hawaii, and by grant funds under P.L. 480 in Israel and Italy. Fundamental research is conducted on fruit constituents that are involved in the flavor, color, and texture of fruit products, and includes development of laboratory tools to isolate and characterize individual components, investigation of such components as they occur naturally and as they are altered by operations involved in preservation, and the relationships between the components and the product values being preserved. Applied research is conducted to develop new and improved processes and products that will increase the utilization of fruits and tree nuts, including the development of high quality concentrated and dehydrated products and more stable shelled tree nuts and the selection of improved processing varieties.

The Federal program of research in this area totals 28.5 professional man-years. Of this number, 10.7 are assigned to chemical composition and physical properties (including one employee whose salary is provided by the Dried Fruit Association of California); 2.4 to new and improved food products; and 15.4 to new and improved processing technology (including 0.5 professional man-years support for an employee whose salary is provided by the Dried Fruit Industry Research Advisory Committee, whose membership represents the California Raisin Advisory

Board, the Dried Fig Advisory Board, the California Prune Advisory Board, and the Dried Fruit Association of California and 0.5 professional man-years support for an employee whose salary is provided by the Diamond Walnut Growers, Inc.). In addition the Division sponsors 4.0 professional man-years of research under P.L. 480 including 1.0 on basic studies which are part of an investigation also concerned with vegetables, and 3.0 on applications of research findings.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 11.2 professional man-years divided among subheadings as follows: Chemical composition and physical properties 4.5, new and improved food products 2.2, new and improved processing technology 4.2, and new and improved industrial products and feeds from by-products of fruit processing (including waste disposal) 0.3. Fundamental studies are conducted on the components concerned with texture, color, and flavor properties of processed fruits and tree nuts. New fruit and fruit juice products and processes are concerned with novelty and convenience factors of importance in developing new markets. Blanching, freezing and dehydration processes are investigated. A fruit softening problem with brined cherries is studied cooperatively with Department of Agriculture scientists.

Industry and other organizations including food processors and distributors, industry and trade associations, and allied industries and suppliers conduct research programs that are predominantely concerned with specific applications to individual corporate problems. A portion of the research of processors involves the extension to commercial status of new processes of products that have been developed by the Department of Agriculture or other public or trade-sponsored agencies. A limited amount of oriented basic research is conducted by trade associations and the supplier trade, including sanitation in the food preservation industry, chemical residues and their toxicity limitations, packaging materials, and fundamental descriptions of raw materials used in food processing. Estimated annual expenditures in this area are equivalent to approximately 200 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Flavor Components. Of great importance to flavor chemistry in recent years has been the application of gas-liquid chromatography techniques. High instrument sensitivity was achieved by use of dual columns with dual-flame ionization detectors and programmed temperature control, so that it is now possible to separate and make tentative identifications of important volatile components from the

headspace atmosphere of a small flask containing only a gram or two of the food product under investigation. The procedure, developed at Albany, California is sufficiently distinctive to warrant a special name. The instrumental record developed by the new procedure is called an "aromagram." Earlier extraction and concentration procedures are not required so that possibility of introducing artifacts is minimized. Sample size required is very small and food samples can be withdrawn from processing lines at different stages or from packages during storage experience and analyzed for compositional changes that may be correlated with subjective evaluations of products. The aromagram procedure is more sensitive by a factor of ten than any method previously used. However, while this is below the sensory threshold of many flavor components, it is not sensitive enough to detect some odorous compounds at levels detectable by the human nose. Only a beginning has been made in using the aromagram in fundamental studies of fruit flavor. Characteristic differences in composition of storage-deteriorated frozen strawberries from those stored at protective low temperatures have been observed. Further applications of the aromagram procedure to raw material quality control for processing, changes induced by processing, and storage deterioration of processed products have been initiated. Developments are continuing to increase even further the sensitivity of equipment to measure flavor components.

2. Pigments. The preservation of natural fruit color is frequently a most difficult accomplishment. Generally, pigments are altered by processes used or degraded during subsequent storage of the product. The anthocyanins (reds, blues, purples, etc.) are sensitive to acidity changes and, in the naturally acid fruits, they tend to be bleached or adversely modified during heat treatments and during subsequent storage of processed products. Chemical structure and the nature of instability of anthocyanins have been studied. Natural and synthetic anthocyanidins have been prepared in which one or more of the hydroxyl groups have been stabilized by methylation. Thus, the principal locus of instability in natural red pigments has been detected. By methylation of the 3-position hydroxyl on pelargonidin, a bright red color was retained for two weeks whereas the unaltered compound was decolorized in two hours by sulfur dioxide. The chemical structural changes of such pigments with pH change has been rigorously investigated. It was found that at pH 3 to 4, one of the ring structures opens to give the corresponding, almost colorless, chalcone. Ring-opening is not favored if the compound contains a methoxyl or glycosidoxyl grouping at position 3. This clear understanding of the nature of the instability of this type pigment could lead to important applications to reinforce the natural red pigments of fruit juices, preserve the natural color of canned fruits, and restore the natural color of fruits, such as cherries which have been decolorized during the preserving process. Preliminary investigations on such applications have been initiated. In addition, the yellow pigments (carotenoids) of Thompson seedless grapes, Italian prunes, cranberries, blueberries,

blackberries, and black figs have been under intensive investigation. These pigments also have important roles in the color quality of processed fruits.

3. Enzymatic Browning. More effective ways are sought to prevent the brown discolorations of fruit caused by chemical reactions catalyzed by naturally occurring enzymes. The fundamental approach to this objective has been to understand the chemistry of chlorogenic acid (the principal substrate of the enzymic browning) and find methods for altering this and similar substrates. The action of a bacterial species (Pseudomonas fluorescence) degrades chlorogenic acid to non-browning components. Further investigation led to use of purified liver enzyme (transmethylase) to modify the darkening components in apples to prevent discoloration, and then it was discovered that natural enzymes occurring in apples would accomplish the chemical modification if conditions were adjusted properly. Thus, it is possible to adjust the pH of apple slices to 8.0, allow the enzymatic transformation to take place, adjust back to a normal pH, and obtain an apple slice that is not subject to enzymatic discoloration. Other fruits and vegetables exhibiting enzymatic darkening are being introduced into such investigations in searching for further applications. Furthermore, the extension of this procedure for apples to commercial operations is being explored.

4. The Chemistry of SO_2 in Dried Fruit. Several of the dried fruits are treated with sulfite (SO_2) in order to preserve prime quality and stabilize vitamins A and C. In subsequent storage of these products, the sulfite level drops, and in long shipments and harvest-to-harvest storage it may drop below the effective level. Furthermore, for export shipments, a high initial level may be required to allow for the exigencies of the marketing operation. In several of our important overseas markets, limitations on the level of sulfite are restrictive and it is necessary to learn more than is now known about the fate and mode of action of sulfite so that its use can be improved or alternative methods can be developed to retard deterioration in these products (pears, apricots, golden raisins, apples, and peaches). The main pathway for sulfite loss has been found to be oxidation to sulfate, and much of the loss occurs in the dehydration or sun-drying of fruit. Light accelerates the oxidation so that certain dehydrated products (particularly apricots) will retain sulfite better than sun-dried products. The better retention can be equated to lower required concentration of sulfite. It is not possible to achieve the sun-dried quality by conventional dehydration although a new procedure is being developed that shows excellent promise (paragraph C,2, of this report). Effects of storage temperature, humidity, and type of packaging for dried fruits on the retention of sulfite are being studied. In general, high temperature, high humidity, and transparency of package are associated with high sulfite disappearance rate. Sulfite acts in the role of an antioxidant in

preventing non-enzymatic browning in dried fruits. It may also have other modes of action, as in the bleaching of anthocyanin pigments, complexing with reactive sugars, and the control of enzymatic discolorations. These functions of sulfite are being investigated.

5. Texture of Fruits. Little is known of the enzymes involved in the formation of cell wall polysaccharides of plant tissue. This, in part, is due to the dearth of knowledge of the cell wall constituents themselves. Work in this complex field has been initiated, using an enzyme preparation obtained from germinating mung beans. By use of radioactive tracer techniques, the enzyme preparation was found capable of forming two polysaccharides from glucose and fructose. One of the polysaccharides was soluble in hot water, the other not. The former is apparently formed by a previously unrecognized enzymic pathway with the glucose incorporated into the polysaccharide, intact. This polysaccharide is unusually resistant to acid hydrolysis. This study has further offered a clear insight into the nature of an alternative pathway of glucose metabolism bringing a new enlightenment to the way higher plants utilize carbon sources for cell wall synthesis.

6. Ascorbic Acid Biosynthesis in Plants. L-ascorbic acid (Vitamin C) occurs in all higher plants but little is known of its biosynthetic origin, its function, or the nature of its accumulation in fruits and vegetables. The enzymes involved in these processes are being studied by radiotracer techniques in which precursors of ascorbic acid are labeled with C^{14} and fed to living plant tissue. Ascorbic acid, later isolated from the plant, demonstrates by the amount and pattern of its radioactivity the pathways by which the vitamin is formed. It has been proven that glucose is converted, in strawberries, to ascorbic acid and not to an isomer, D-arabo-ascorbic acid as suggested by other researches. The net result of this has been to demonstrate that plants possess the astonishing capacity to convert a D-sugar to L-ascorbic acid by changing the configuration about a single carbon atom. Interrelationships are being elucidated among glucose, galacturonic acid (the main building block of pectins and important in textural quality of fruits), methyl ester of galacturonic acid (an important compound in the textural quality of fruits), and ascorbic acid. The methyl ester of galacturonic acid is a better precursor of ascorbic acid than is the acid itself. On the other hand, both the acid and its ester function in pectin synthesis and both undergo reduction to the same compound, galactonic acid.

7. Chemical Attractant for Fruit Flies. Drosophila (vinegar fly, fruit fly) is a serious pest in the fruit processing industries. When infestations are high in orchards and vineyards and around processing plants, it becomes difficult to pack products substantially free of insect fragments and eggs. Insecticide sprays now in use are difficult to control so as to avoid inadvertent residues in

products, and processors would prefer to use baited traps or other means of control if such were effective. A cooperative project between the Western Utilization Research and Development Division and the Entomology Research Division was conducted to explore the chemistry of possible attractants for Drosophila and to develop better baits for the fly. Preliminary studies were only partially successful and the project has been terminated for the present time. Juices and purees from ten different fruits (strawberry, Scarlet grape, Thompson grape, Italian prune, French prune, apple, peach, Mission fig, Kadota fig, and Bartlett pear) were fermented under various conditions and times and the products of fermentation were separated. These products were subjected to screening as attractants for adult Drosophila by the Entomology Research Division, Beltsville, Maryland. About 1,000 samples were tested. None of these proved to be as attractive as the yeast-sugar-water-vinegar mixture used as the standard lure. Nor were trends noted which suggested concentrating effort on particular fractions.

A commercial product has been placed on the market and is understood to be an effective bait and poison for Drosophila control. In view of the availability of a commercial, effective attractant and of the absence of promising leads from the work already done, this project was discontinued.

8. Preservatives for Dried Fruit. Present market practice involves a tenderizing treatment for retail packs of dried fruit. Prunes, figs, and dates are sold at moisture contents that will allow growth of mold and yeasts unless preservatives are added. The industry has depended upon ethylene and propylene oxides as antimycotics but the 1958 amendment to the Pure Food and Drug Act required proof of nontoxicity of residues from such treatments. Methyl and ethyl formates, used mostly for insect control in raisins, also require clearance. Pharmacological tests were conducted, supported in part by the dried fruit industry who provided the salary of a collaborator, to determine the toxicity information necessary for clearance of these preservatives by the Food and Drug Administration. Propylene oxide and methyl formate have now been approved for use as preservatives for dried fruits. Studies on ethylene oxide are nearly completed, and approval will depend largely on the legal interpretation of the Delaney Clause of the Food Additive Amendment. This clause rules out additives whose use in any proportion in animal diets can cause cancer. Ethylene oxide has not been demonstrated to be carcinogenic but, when fed in large quantities, can cause oxalate stones in rat bladders. The irritation of the stones has led to bladder tumors in the experimental animals. Thus, at levels below which stones would be caused, ethylene oxide could not cause cancer and should be cleared. Because of the secondary effect at high levels in the diet, it remains to be decided whether ethylene oxide will be allowable as an additive to dried fruits under the Delaney Clause. As described

elsewhere (paragraph C,3, this report) another antimycotic has been found effective for dried fruit and the industry problem is now largely resolved. Investigations on the toxicity of preservatives for dried fruit will soon be completed and the industry support may be transferred to other critical problems in the processing of dried fruits.

9. Microbial Flora in Fruits and Vegetables. Fundamental studies on microbial flora within the tissues of fruits and vegetables have been conducted under P.L. 480 in the Department of Food Technology, Agricultural Research Station of the Ministry of Agriculture, Rehovot, Israel. Fruits have been found containing viable micro-organisms within the tissues. It is probable that these organisms gained entry during the formation of the fruit on the plant, but this has not been conclusively demonstrated or proven. Samples studied included grapes and olives and, also, grapefruit and orange. Bacteria of the Xanthomonas, Pseudomonas, Enterobacteria, and Corynebacteria groups have been found, as have yeasts of the Nematospora. Such entrapped micro-organisms can make only a limited development and grow rapidly only when the tissue has been disturbed by injury or maceration. The relationship of such adventitious microbes to processing quality of fruits has not been revealed by these studies and may not yield itself to techniques that are currently available.

10. Enzymes Involved in the Ripening of Fruits with Ethylene. The effects of ethylene gas on maturation rates of harvested fruits in storage have been known for some time. The knowledge has served as a basis for controlled ripening of lemons and bananas, and accumulation of ethylene has an adverse effect on fruits to be held for long periods in cold stores. Basic research is under way to learn the biochemical nature of the ethylene effect on maturation of fruits so that its function can be better controlled and made more useful. The avocado was selected as an example fruit to study because it is available the year around, is a good producer of ethylene, and has a strong response to ethylene treatment. Avocados are exposed during maturation to ethylene synthesized with radioactive carbon or hydrogen. The labeled ethylene reacts in the maturing fruit and analyses are made to determine the reaction products. When avocados were ripened in the presence of hydrogen-labeled ethylene, 15% of the label was found in the hydrocarbon, toluene. The establishment of this hydrocarbon as a major reaction product indicates an unusual, perhaps unique, metabolic pathway involved in maturation.

B. New and Improved Food Products

1. Fruit Powders. Preliminary work with the newly developed foam-mat drying technique has demonstrated the possibility of producing powders from applesauce, peach, pear, and apricot nectars, pineapple juice, berry products, Concord and wine grape juices, prune puree and whip, and plum puree. Enzymic flavor release methods have been studied to seek the fuller development of fruit flavor in dried fruit powders. In preliminary studies with apples, volatiles were produced. This aspect of the product development will continue. Subjective evaluations of products are conducted to seek improved quality, particularly improved flavor of products.

2. Dehydrofrozen Fruit. Laboratory studies are completed on the development of processes to partially dry fruits and preserve them by freezing. Extensive commercial operations now exist in the upstate New York area (estimated production in excess of 8,000,000 pounds of product per year of dehydrofrozen apples for the bakery trade). During this reporting period, cooperative work has been conducted with several processors and with the New York Agricultural Experiment Station to adapt processes developed with Western grown fruits to local varieties and to adapt processes to the different types of equipment installed in the plants. Interest in dehydrofreezing cherries is high and technical problems are still under investigation. The Albany, California staff will continue to cooperate with the Experiment Station and industry in further extension of this technology but no laboratory research is contemplated.

3. Processing Quality of Northwest Soft Fruit and Berry Varieties. Canning, freezing, and preserving of new and improved berry and tree fruit varieties have continued in field stations at Prosser and Puyallup, Washington in cooperation with horticulturists of the Washington State Agricultural Experiment Station. Selection of varieties to resist disease and improve yields is a task without end. Processing quality must also be considered for most fruits, because an increasing proportion of the harvest is preserved by one means or another. (About 90 percent of Northwest strawberries are processed.) Decreasing production of the Marshall strawberry has emphasized the need for a new variety, with good quality for jam and preserves. Processing studies at Puyallup have helped establish the Cascade variety for this purpose. Incursions of root-rot in raspberry plots have emphasized the need for resistant varieties. Expansion in blueberry and rhubarb production depends on improved varieties and processes and new products to build new markets. Strawberries with a growth habit that will make them easier to pick and thus reduce harvest labor costs are becoming more needed as each year passes. Apricots, peaches, and plums with better texture, flavor, and color and with different maturation character to extend the harvest season

are needed. Processing aspects of quick-decline-affected pear orchards may have important economic impact. These problems are subject to continuing investigation in cooperative studies. Each harvest season, new varieties are processed as well as the standard commercial varieties, with which they are compared. Several years and many replicated plots are required to establish the value of a new variety under varied conditions and make a fair comparison with the established varieties. Then, after careful evaluation the horticulturists may release a very small fraction of the numerous trials, with a complete statement, including the processing character for various uses (canned, frozen, juice products, and preserves).

4. Maturity Index for Harvesting Fruit. An index to measure maturity is necessary for many fruits if prime raw material is to be available for processing. Control of quality of processed products starts with the quality of the raw fruit and optimum development of color, flavor, and texture on the tree is very important. Soluble solids and acid measurements have not provided adequate indexes for some fruits. Fresh fruit color for purple plums and dark sweet cherries appears to be a more useful index of when the fruit should be picked but visual examination of color is not accurate enough to be depended upon. The optical density of an extract of sample fruit can be accurately measured with readily available colorimeters and has been found to be the basis for predetermining the color of canned fruit. Extension of this new maturity index to commercial canning operations is beginning and is expected to provide improved products for marketing these fruits.

5. Canned Concentrated Peach and Apricot Purees. Studies on the effects of temperature and temperature variations encountered in transportation and distribution of concentrated peach and apricot purees as a basis for improving processing conditions have been conducted under a P.L. 480 grant to the Experiment Station for the Food-Preserving Industries, Parma, Italy. Extensive analyses and evaluations of color, flavor, aroma, sugars, nitrogenous constituents, volatile and non-volatile acids, and vitamins have been conducted, comparing single strength and concentrated purees. Processing effects have been observed related to the organoleptic values of products but concentration has been accomplished with no important change in nutritive components. This project will terminate soon.

C. New and Improved Processing Technology

1. Foam-mat Drying. Over 50 agricultural commodities have been successfully dehydrated by the foam-mat drying process invented by Department engineers at Albany, California. These include applesauce and juice, peach, pear, and apricot nectars, berry products,

Concord and wine grape juice, and prune puree and whip. Continuous, automatic equipment has been designed and constructed for continuing experimental studies. A commercial dryer of similar design has been installed in a food processing plant in California and several other industry applications of this novel dehydration method are being investigated with pilot operations to develop commercial-scale equipment. Most products tested can be dried at atmospheric pressures without off-flavor development or discoloration. There is a loss of volatile flavor components. A major problem in the technology of each product so dried is to find means for adding back or redevelopment of flavor. The enzymic release of volatile flavor from non-volatile precursors retained by dried apple products has shown promise in preliminary investigations. Solid carriers, such as sugar and other carbohydrates are used to "lock in" volatile flavor components and equipment has been designed to mix and form the carbohydrate-flavor mixture. This equipment operates continuously and has advantages over previous methods by minimizing heat requirements, thereby minimizing chemical alteration of the volatile flavor components. Improved foam stabilizers, required for some products in foam-mat drying have been selected or developed to improve product quality. Drying cycles have been determined for specific products to prevent serious quality loss during processing and to increase heat and moisture transfer and mechanical efficiencies so that processing costs may be reduced.

2. Dried Fruit Processes. Problems encountered in the conventional sun-drying of fruits include occasional product losses due to inclement weather and contamination during field exposure. Sun-drying of cut fruits produces a desirable translucency in the product and is the lowest cost method. If the quality factors could be made equal, many producers of dried fruit would welcome a dehydration procedure that would be used when conditions beyond their control force them to curtail sun-drying operations. An improved process, called "Dry-Blanch-Dry" or "DBD" has been developed in which dehydrated apricots, peaches, pears, and raisins have been produced with "sun-dried" quality. Commercial trials were run comparing DBD with sun-dried apricots and subsequent stability tests made of the two products. An important extra credit for the new process was observed in that sulfur dioxide disappearance rate from the product was lower for the DBD. This suggests the possibility of using somewhat reduced sulfur dioxide levels to achieve the same stability, a factor of importance in exportation of dried fruit because some countries restrict the amount of this stabilizing additive to levels where product quality may be jeopardized in the long channels of trade. The DBD process involves a partial drying of the prepared fruit, followed by steam blanching and finish drying. With apricots, peaches, and pears, the halved, pitted fruit is sulfured as with sun-drying prior to the first drying step. In addition, bin-drying procedures for the final moisture removal (from 16% down to

12%) from cap-stemmed muscat raisins were established in pilot operations. Equipment cost savings of over 50% by using bin-drying instead of conveyor-belt dryers are estimated.

3. Stabilizing Dried Fruit. Research on procedures for stabilizing dried fruits is conducted with support of the Dried Fruit Association, which provides the salary of a scientist assigned to this project. Critical studies of the effects of temperature, humidity, exposure to light, and moisture content on the stability of prunes, figs, dates, apricots, and apples have been completed. Methods for determining product moisture content were evaluated, and suitably reproducible methods were established for use in this investigation. The question of permissibility of ethylene and propylene oxides as antimycotic agents for tenderized prunes and figs led to a search among additives already cleared for food use by the Food and Drug Administration. A process was developed to apply potassium sorbate by spraying a water solution. Spray-treated, high-moisture prunes and figs resisted microbial spoilage and this process is now standard in the industry with all but one major dried fruit producer using it. Approximately 70,000 tons of dried figs and prunes are given this treatment annually. A necessary analytical method for determining sorbic acid and sorbates in dried fruits was developed. Two additional processes relating to this study were developed in the laboratory and are undergoing trials to determine commercial applicability. First, a novel method for rehydrating dried fruits for tenderizing. Dried fruit pieces are heated in steam and then dipped in cold water, which induces remarkably rapid imbibition of water. Second, a simple heat treatment was discovered to prevent the "setting up" of ground-up raisins (raisin paste--a product used in the baking trade). The ground-up product is held at 120° F. for 2 days and it will then remain spreadable and of a satisfactory consistency for remanufacturing use. Without the mild heat treatment the product must be reground to be used.

4. Wax-coating Raisins. When raisins are added to breakfast foods or similar products, the dry cereal ingredients draw moisture from the raisins making them hard and unattractive. For such products to retain quality long enough for orderly marketing, a barrier must be used to reduce the rate of moisture transfer. Beeswax is a suitable barrier but difficulty has been encountered in the past in applying it so as to have a uniform, continuous coating and, at the same time, reduce the amount added to an economical level--about 2-1/2% by weight of wax, or less. A machine capable of applying such a thin coating of beeswax has been designed and constructed. Preliminary tests indicate that the machine will do an exceptionally good job with wax contents ranging from 2% for large raisins to 2-1/2% for small raisins. Such coatings are effective in slowing the moisture loss from raisins, are not objectionable from a flavor standpoint,

and are in the economical range specified by the breakfast cereal industry. Commercial trials of the new equipment are being conducted.

5. Softening of Brined Cherries. Occasional outbreaks of a serious softening condition of brined cherries has plagued the fruit industry of the Northwest. Cooperative studies have been conducted with the Oregon and Washington Agricultural Experiment Stations to determine the cause of such outbreaks and to develop methods for control. Studies of natural fruit enzymes and enzymes produced by microorganisms were studied in regard to softening. Modifications of brine formulas and use of various additives were also investigated. Using laboratory procedures in which softening was induced in sound cherries, a number of tentative factors were established including the inhibitory effect of calcium, and the softening effect of a number of fungi inoculum. Brined cherries to which pectin-splitting polygalacturonase had been added softened, yet naturally softening cherries have been observed in which this enzyme could not be detected. Treatments that have been effective in laboratory tests, including excess calcium salts and one of the common detergents, have not been effective in controlling softening in a commercial sample of cherries undergoing deterioration. Heating the fruit and brine to pasteurization temperatures has so far been an effective method of control. Although the problem of brined cherry softening has not been resolved, the texture testing procedures developed as necessary for the conduct of research, have been picked up by the industry as a useful quality control tool to provide advanced indication of a softening "infection" in commercial operations.

6. Stabilizing Shelled Nuts. Shelled tree nuts are a convenient food item, but they tend to darken and turn rancid rather quickly. An investigation of factors involved is supported, in part, by the Diamond Walnut Growers, Incorporated, which supplies the salary of one chemist assigned to the field station at Pasadena, California. Previous work at Pasadena resulted in a process that has had extensive commercial use--stabilization was achieved by critical control of moisture content, coating the kernels with antioxidants, and sealing in a package of transparent film with a very high moisture barrier rating. Less expensive packaging is required to maintain shelled nuts in a more attractive competitive position. Chemical and technological studies are in progress with walnuts to determine the constituents involved in dark discoloration and oxidative rancidity development and to develop methods of controlling the deleterious effects. Constituents are identified and changes they undergo under various conditions of temperature, light, and atmospheric gas are measured. Correlations of constituents with discoloration and rancidity development are being developed. A project along similar lines, but with macadamia nuts, has been initiated under contract with the University of Hawaii. Macadamia nuts appear to

to be a useful crop to replace acreage of pineapples, in excess production, and coffee, a product in some distress because of high production costs and difficult quality controls.

7. Northwest Grape Juice. Processing of Concord grape juice and the effects of cultural practices on composition and quality of juice are under cooperative investigation in Prosser, Washington with the State Agricultural Experiment Station. Organoleptic quality of this grape juice has been determined to be most closely related to the soluble solids and acid content. Over a substantial range of difference in naturally occurring methyl anthranilate (considered to be the principal flavor component of Concord grapes) flavor differences were not detected. Juices from high and low soluble solids grapes could not be distinguished from each other after adjustment to the same soluble solids and acid content. This indicates that cultural practices giving maximum yield can be used even at the expense of producing grapes of lower soluble solids. Low solids content would have no effect on product quality in juices packed as frozen concentrate because refined sugar and acid are added during processing to such concentrates in any case. Furthermore, Concord grapes were found to tolerate a rather wide range of heat treatments during extraction and processing without appreciable alteration of flavor. Thus, in commercial operations it is possible to make considerable adjustment to provide better extraction of color and flavor components.

8. Fouling of Heat Transfer Surfaces. Fundamental engineering studies were conducted on the nature of fouling of heat transfer surfaces in evaporators used in the concentration of fruit juices and purees. Fouling was increased by increasing surface temperature, vapor fraction in the tubes, and product viscosity. Furthermore, fouling was markedly more rapid when products were being warmed compared to when they were being boiled. A resistance-impedance thermometer concept was developed to obtain accurate surface temperature and related heat transfer measurements. This can be extended generally to heat transfer investigations and operations control in the field of temperature-sensitive fluids. Observations of this research lead to conclusions that can influence design and use of evaporators. Studies have been initiated to determine the feasibility of such applications. For example, it could be predicted that vapor fraction would be reduced by using upflow instead of downflow in an evaporator tube. Experimental work confirmed this prediction and, within the practical operating range studied, upflow resulted in only half the fouling rate of downflow. Studies of fouling in tubes for warming liquids have not been conclusive yet because of difficulties in measuring true liquid temperature at various points in the test system. A new electrical warming system has been designed to replace the steam heat source. Voltage drop in the system can be measured

to represent heat liberated and the average liquid temperature calculated by heat balance. Fouling data to guide design of long-tube evaporators is thus being obtained.

9. Continuous Juice Press. A new, larger, but still experimental continuous press was successfully plant tested as a preliminary dejuicer during the 1960 season. When operated on firm apples such as Newtown Pippins, the 5' x 1' machine had a capacity of a little over 6 tons an hour, for a gross juice yield of about 150 gallons per ton of fruit. With ripe, mealy Red Delicious apples the capacity dropped to about 4 tons an hour, with a gross yield of approximately 120 gallons per ton. Secondary pressing is essential for yields comparable to those obtained in a good rack-and-cloth operation (170-180 gallons per ton). Attempts were made to use a screw press for this purpose. The repressing operation was successful only when a substantial amount of wood pulp was added to the ground fruit--about 3/4 of 1% by weight for firm fruit; 1-1/2% for ripe fruit. Combined juice yield from the two machines was about 180 gallons per ton, a figure competitive with most rack-and-cloth operations. Net yield, after Pectinol treatment, was about 145-150 gallons per ton, compared with 158-162 gallons per ton for the rack-and-cloth press. Observations were made of commercial attempts to produce apple juice of adequate yield and freedom from suspended solids. Although some equipment did a fairly good job when apples were in the right condition, results were not so good when the raw material quality changed as it will inevitably do during the storage season. A conclusion was reached that more basic knowledge of the pressing characteristics and processing requirements of apples of different varieties, maturities, and composition is needed to serve as a basis for further developmental work on a practical continuous dejuicing system.

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Area 7 POTATOES--PROCESSING AND PRODUCTS--WESTERN LABORATORY

Problem. The potato industry, faced with a continuing decline in the consumption of fresh potatoes, is becoming more and more dependent upon the development of new and improved processed products to maintain markets and to avoid recurring economic disasters. Crop perishability, supply fluctuations, and the inelasticity of demand, result in wide swings in price with even slight surpluses. In producing areas having a substantial processing industry, depressive lows are moderated by advance contracting by processors prior to harvest. However, in many important potato growing areas processing has not yet developed, and vulnerability not only still exists, but is exaggerated by the growing competition of processed potato and other competing food products. A continuing improvement in processed potato products is clearly required if processing is to expand fast enough to offset the progressive decline in use of fresh potatoes.

To make possible required improvement in quality of processed potatoes, ways must be found to eliminate the stale, "earthy," rancid, "green," and "warmed-over" flavors that are sometimes encountered in potato products, including dehydrated mashed potatoes, dehydrated diced potatoes, frozen French fries, frozen patties, and potato chips. Equally important, methods must be devised for retaining the desirable natural flavor of the freshly cooked potato in the processed product. Recently developed research methods offer an opportunity to isolate and identify the chemical constituents responsible for the natural flavors and the off-flavors, to develop rapid and sensitive analytical methods for their measurement, and to determine the raw material factors controlling formation of the various desirable and undesirable constituents in the fresh potato. Further improvement in the texture of potato products is also needed. Fundamental histological and chemical investigations could be used to determine the causes of differences in the textural character of potatoes, as a basis for developing improved processing methods. Enzymes play a great part in controlling the entire compositional pattern of the potato, not only the constituents responsible for flavor, off-flavor, color, and texture, but also those responsible for disorders such as "black spot." "Black spot" causes severe losses both to those who market potatoes in fresh form, and to those who process potatoes, because trimming costs are sharply increased and product yields reduced. Greatly increased knowledge of enzymes is needed as a basis for solution of the black spot and other problems, to increase use of potatoes by reducing costs, and to improve quality of both fresh potatoes and processed potato products.

USDA PROGRAM

In the Western Utilization Research and Development Division, basic and applied research on potato products is conducted at the Division headquarters at Albany, California, and by grant funds under P.L. 480 in England. The chemistry of potato flavor and the compounds involved in deterioration of potato products are studied to provide a basis for new and improved potato processes and products. Histochemical studies are conducted to elucidate factors involved in the texture of potato products. Basic investigations on the enzyme systems involved in potato product discoloration and the role of sulfur dioxide in preventing non-enzymatic browning are in progress.

The Federal program of research in this area totals 7.6 professional man-years. Of this number, 3.6 are assigned to chemical composition related to flavor, color, and texture of potato products (including 0.5 professional man-year's support for a chemist whose salary is provided by the Instant Potato Granule Manufacturers Association), 0.5 to new and improved products based on dehydrated potatoes, and 3.5 to technological and engineering research on processing methods. In addition, the Division sponsors 6.6 professional man-years of research under P.L. 480 on basic studies.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

Related program of all State Experiment Stations and industry and other organizations is reported by the Eastern Utilization Research and Development Division in Summary of Current Program and Preliminary Report of Progress.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition Related to Flavor, Color, and Texture of Potato Products

1. Flavor Components. Of great importance to flavor chemistry in recent years has been the application of gas-liquid chromatography techniques. High instrument sensitivity was achieved by use of dual columns with dual-flame ionization detectors so that it is now possible to separate and make tentative identification of important volatile components from the headspace atmosphere of a small flask containing only a gram or two of the food product under investigation. The procedure, developed at Albany, California, is sufficiently distinctive to warrant a special name. The instrumental record developed by the new procedure is called an "aromagram." Because extraction and concentration procedures are not required, the introduction of artifacts is minimized. Sample size required is very small; food samples can be withdrawn from processing lines at different stages or from packages during storage experience and analyzed for compositional changes

that may be correlated with subjective evaluations of products. The aromagram procedure is more sensitive by a factor of ten than any method previously used. However, while this is below the sensory threshold of many flavor components, it is not sensitive enough to detect some odorous compounds at levels detectable by the human nose. The aromagram method was developed, in part supported by the Instant Potato Granule Manufacturers Association. The method makes it possible to measure the concentration of certain compounds which are the result of oxidative deterioration (rancidification) of potato products, and of others which are the result of non-enzymatic browning (scorching). It is thus possible to measure objectively and quantitatively the degree of these two types of flavor change in any sample. Study of the various processing operations to determine their effects on product flavor is now possible on a rational basis and can be expected to lead to improved processes and products. Investigation of the chemistry of storage deterioration of products is likewise in progress, using the most advanced methods for measuring components.

2. Enzymatic Browning of Processed Potatoes. Fundamental studies on the enzymatic browning of potatoes are in progress at the Low Temperature Research Station in Cambridge, England, supported by a grant under P.L. 480. Browning does not occur until the cellular structure is damaged, as by peeling or bruising the potato and, perhaps, under some disease conditions. The nature, distribution, and mode of action of enzymes responsible for this type of brown discoloration of potatoes and potato products are investigated by several approaches. Relationships have been found between browning tendency and potato variety, location where grown (possibly cultural conditions), concentration of enzymes (tyrosinase and polyphenol oxidase), and concentration of the chemical substrates upon which the enzymes act (tyrosine and chlorogenic acid). A direct correlation has been found between concentration of one of these substrates (tyrosine) and enzymatic browning tendency, and the amount of tyrosine was found to be inversely correlated with the total solids content of potatoes. Factors that affect the tyrosine content in potatoes will now be investigated including the effect of variety, soil and fertilizer use, and climatic conditions. Related studies with one of the enzymes (polyphenolase) from other plant sources have been conducted to obtain a basic foundation for understanding the chemical and metabolic reactions involved in the phenolase-discoloration system.

3. The Role of Sulfur Dioxide in Dehydrated Vegetables. A fundamental investigation of the chemical fate of sulfur dioxide or sulfite in dehydrated vegetables is being conducted at the Covent Garden Laboratory in London, England supported by a grant under P.L. 480. The objective of this research is to determine the chemical mechanism through which sulfite exerts its protective action on dehydrated vegetables. Model chemical systems have been used in which the effects of sulfur dioxide and other components have been observed as they

affect simple browning reactants (e.g., glycine and glucose), and other more complicated chemical reactants (e.g., citral in place of glucose). By use of sulfur-35 as a radioactive tracer, the chemistry and migration of sulfite applied to potatoes during dehydration have been studied. By these means some of the complications of the mechanism through which sulfur dioxide prevents non-enzymatic browning are beginning to unfold. A common chemical structure in glucose and citral (the carbonyl group) is involved in a reaction with amino acids, such as glycine, as a primary step in forming the brown pigment. Citral is more reactive than glycine because of its unsaturated structure. Sulfite seems to block browning by prior reaction with the carbonyl, but unsaturation, as in citral, can be responsible for a rapid migration of the sulfite within the molecule, allowing a more rapid color formation by freeing the carbonyl. Effects of various reactants, calcium, iron, phosphate, and ascorbic acid (Vitamin C), on rate of browning are being elucidated in this study.

4. Potato Components Related to Cooking Texture. Contract research at the University of Idaho was completed, in which 69 lots of potatoes over a two-year period were analyzed. In addition, each lot was cooked by a standard procedure and evaluated by a rapid method developed to evaluate potato texture. The texture of cooked potatoes was positively correlated with total solids, alcohol-insoluble solids, starch concentration, total ash, and alkalinity of ash; it was not correlated with content of pectin, crude fiber, galactose, arabinose, calcium, magnesium, or phytate phosphorus. These findings support a conclusion that potato texture is closely related to starch content and that the composition of the cell wall and supporting tissue structures is not of major significance.

5. Potato Enzymes. A limited project was completed on enzymes concerned with sugar conversions in potatoes stored for processing. For the first time it was unequivocally demonstrated that potatoes contain an enzyme (uridine diphosphate glucose-fructose transglucosylase), which synthesizes sucrose (common sugar), and another enzyme (invertase), which removes sucrose by hydrolyzing it to the reducing sugars, fructose and glucose. Enzyme extracts from potatoes stored at low and high temperatures had equal sucrose-synthesizing activity. However, low temperature potatoes had three to four times the sucrose-splitting activity. Much more of the sugar-synthesizing enzyme was found in California White Rose variety potatoes, which tend to have a high sugar concentration, than in Russet Burbanks, which are low in sugar-forming tendency.

6. Antioxidant Activity of Polyphenolic Compounds. A fundamental investigation of the chemical mechanism of polyphenolic and other plant constituent antioxidants for use in enhancing the stability of food products, has been conducted at the Aberdeen, Scotland, Research

Establishment and Experimental Factory, sponsored by a grant under P.L. 480. The objective has been to develop new or improved stabilizers that will preserve the quality and improve consumer acceptance of processed foods. The investigators obtained information on the relation between the structure of polyphenols and their antioxidant activities. Such information is of a very basic nature and should be valuable in the extraction of natural plant constituents and their chemical alteration to provide practical antioxidants which combine the ideal chemical structural features with commercial availability. The closing of the Aberdeen laboratory by the British Ministry of Agriculture made necessary the premature termination of this research grant.

B. New and Improved Products Based on Dehydrated Potatoes

1. Product Developments. Investigations on new potato products are conducted, closely connected with technological and engineering research (see paragraph C,1). The use of existing potato products as ingredients of new formulated food products such as instant soups, crackers, and related food items is under consideration on a very limited scale.

C. Technological and Engineering Research on Processing Methods

1. Dehydrated Potato Granules. Alteration of starch in the tissue cells during processing is of primary significance to the textural quality of potato products. Release of gelled starch from ruptured tissue cells of mashed potatoes imparts a sticky or gummy texture. Several methods were found to mitigate this condition by manipulation of the properties of gelled starch in the cooked potatoes. Precook heat treatments and cooking at a controlled temperature below the boiling point resulted in greatly improved products by reducing rupture of tissue cells during processing. Edible food additives, including starch-complexing agents (such as calcium stearate and palmitate) and emulsifiers (such as glycerol monostearate and palmitate), reduce the stickiness of the free starch that is released by rupture of tissue cells. Analytical methods were developed whereby the amount of starch released could be measured in the presence of these additives, which interfere with the chemical method in common use (Blue Value Index). Another textural problem sometimes encountered in dehydrated potato granules is the tendency to incomplete rehydration, which results in a graininess in the reconstituted mashed potatoes. Natural gums, added at levels up to 0.5% of total solids, were found to control this defect. A wide range of conditions and additives were discovered that allow considerable opportunity for manipulation of the textural qualities of dehydrated mashed potato products. Pilot plant equipment was designed and constructed to study effects of various partial dehydration, mixing, and conditioning operations on the quality of dehydrated potato granules. Possible changes

in existing commercial practices were developed within limits of small-scale batch operations. Processing research has now been directed toward study of the unit operations that may adversely affect product flavor, by close coordination of experimental design with the fundamental investigations on flavor components (see paragraph A,1).

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Chemical Composition Related to Flavor, Color, and Texture of Potato Products

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Area 8 VEGETABLES--PROCESSING AND PRODUCTS--WESTERN LABORATORY

Problem. Vegetable crops, in general, are perishable and seasonal and thus, are subject to supply and price fluctuations to the disadvantage of the agricultural economy. In order to expand markets and stabilize prices at a suitable level, new and improved processed products are needed that will be more desirable to the consumer from the standpoint of quality, convenience, stability, nutritive value, safety, and cost. These factors are important for expanding foreign markets as well as domestic. The quality of processed vegetables and the economy of their processing have not improved rapidly enough to increase or even maintain the relative position of vegetables in the American diet, or to increase substantially their contribution to the export trade. The consumption of dry beans and certain other vegetables is limited by the fact that they cause flatulence.

New easy-to-prepare vegetable products are needed, particularly from such commodities as dry beans and peas, which now require hours to prepare. The severe heating required to sterilize low-acid foods, which include most vegetables, seriously impairs the quality of canned products. The stability of all kinds of processed vegetables needs to be improved so that quality and nutritive value will be better preserved during storage and distribution. The safety and effectiveness of new chemical additives, needed to improve the quality and stability of processed vegetables, must be established. Better methods of removing residues of agricultural chemicals from vegetables for processing are urgently needed, as are procedures for decontaminating vegetables exposed to radioactive fallout. Of vital importance is research to reduce the costs of processing in order that the farmer may receive a larger share of the consumer's dollar.

Applied research on these practical problems must be supported by a strong program of basic research on the chemical constituents of vegetables responsible for flavor, color, and texture; on the reactions these compounds undergo before, during, and after processing, on constituents having biological activity; on the microscopic structure of vegetables and vegetable products; and on the micro-organisms which cause spoilage or loss of quality in these products.

USDA PROGRAM

In the Western Utilization Research and Development Division, a broad program of basic research on vegetables and the application of science to new and improved products and processes is conducted at the Division headquarters at Albany, California, in field stations at Pasadena, California and Puyallup, Washington, by contract at Urbana,

Illinois, and by grant funds under P.L. 480 in Rehovot, Israel, and Cambridge and Chipping-Campden, England. Fundamental studies are conducted on the chemistry of vegetable flavor and vegetable pigments, the mechanism of heat resistance in bacterial spores, the composition of dry beans as related to cooking quality and flatulence-producing characteristics, the factors affecting deterioration of dehydrated vegetables, and the microbiology of raw vegetables for processing. Applied research is conducted to develop new and improved products to increase the utilization of vegetables including new, high quality concentrated and dehydrated products and products of improved convenience of use, processes for producing these, and selection of improved processing varieties.

The Federal program of research in this area includes a total of 35.8 professional man-years.1/ Of the total, 22.1 are assigned to chemical composition and physical properties (including 1.0 professional man-years of support for two chemists whose salaries are provided by the California Lima Bean Advisory Board operating under a State Marketing Order; 0.5, support for a chemist whose salary is provided by the National Canners Association; and 0.5, support for two chemists whose salaries are provided by the United States Brewers Association); 3.1 to new and improved food products; and 10.6 to new and improved processing technology. In addition the Division sponsors 9.0 professional man-years of research under P.L. 480 on basic research.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 20.1 professional man-years divided among subheadings as follows: Chemical composition and physical properties, 6.2; new and improved food products, 2.0; new and improved processing technology, 8.6; and new and improved industrial products and feeds from by-products of vegetable processing (including waste disposal), 3.3. Fundamental studies include flavor research on celery, rutabagas, beets, and corn, enzyme inactivation and regeneration relative to food processing, textural firmness of processed vegetables, and other related compositional studies. New and improved food products are developed in connection with characteriization studies of raw material and control of color and flavor deteriorations during processing. Processing studies are concerned with new applications and modifications of various unit operations such as materials handling, enzyme inactivation, preservative treatments, and the like. Limited studies are conducted on handling of processing plant residues to control nuisances, reduce handling costs, and seek development of salable by-products.

1/ Including 1.8 in a contract project on flatulence-producing characteristics of dry beans, initiated during fiscal year 1962 with non-recurring funds from the Administrator's contingency appropriation.

Industry and other organizations, including food processors and distributors, industry associations, and container and equipment suppliers, conduct research programs that are predominantly concerned with specific applications to individual corporate problems. A portion of the research of processors involves the extension to commercial status of new processes and products that have been developed by the Department of Agriculture or other public or trade-sponsored agencies. A limited amount of oriented basic research is conducted by trade associations and the supplier trade, including sanitation in the food preserving industry, chemical residues and their toxicity limitations, packaging materials, and fundamental descriptions of raw materials used in food processing. Estimated annual expenditures in this area are equivalent to approximately 140 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Sulfur-Containing Flavor Components. Fundamental studies are conducted on the chemistry of vegetable flavors to provide a rational basis for improving the quality of processed products. The volatile components of onions have been studied in considerable detail and the chemical nature of onion flavor has been revealed to an advanced degree. Sulfur-containing compounds (disulfides and trisulfides containing methyl and propyl groups) were identified as the principal odoriferous components. Volatile alcohols, aldehydes, and ketones were also separated and identified, but not considered important for flavor. The disulfides and trisulfides were shown to be derived from chemical precursors by rapid enzymic decomposition that occurs as the onion tissue is disturbed by peeling, cutting, and masticating. Flavor precursors in onions were isolated and identified and their enzymic alteration studied. The enzyme involved was isolated and the characteristics of its action elucidated. The enzyme, onion alliinase, hydrolyzes typical sulfur-containing flavor precursors with the liberation of pyruvic acid in an amount proportional to that of the flavor compound formed. Based on this new knowledge, an objective method for measuring onion pungency was developed. A small piece of onion is divided into two portions; one is analyzed directly for pyruvic acid and the other is so analyzed after allowing the naturally-occurring enzyme to react with flavor precursors. The difference in the amount of pyruvic acid is an excellent index of the flavor concentration. This flavor analysis was found to be correlated to a high degree with subjective evaluations of onion pungency. The method is applicable to breeding improved onion varieties for processing where high pungency is required to counteract flavor lost in processing and, also, applicable to the selection of raw material for processing. In breeding research, the analysis can be run on a small sliver of tissue from the onion and, if found to be of desirable quality, the same onion can be used for propagation. Analysis

for the flavor release enzyme can be applied to onion dehydration process studies and production control. Specific operations that lead to destruction of the enzyme should be avoided. Analyses can be run to adjust conditions to minimize such destruction.

Another type of sulfur-containing vegetable flavor is found in cruciferous vegetables (mustard, cabbage, etc.). These compounds, the highly pungent isothiocyanates, are also enzymatically produced from precursor components. A convenient analytical method for measuring the enzyme, myrosin, was developed; and the chemistry of this flavor release has been investigated. It was found possible to enhance certain important flavor notes by enzymic action, in vegetable products whose volatile flavoring components had been dissipated by preservative treatment.

There still remain some factors in onion flavor to be resolved, including the lachrymatory principle and certain sulfur compounds that are more complicated in structure than the ones yet studied (e.g., sulfur-containing peptides containing two or more amino acid components). Furthermore, the extension of enzymic flavor release investigations to other important vegetables remains to be done.

2. Flavor Studies of Peas. Cooperative work supported, in part, by the National Canners Association, which provides the salary of a chemist assigned to the project, is conducted on the composition of peas to provide a basis for technological improvement of canned pea flavor. Large-scale extractions of peas (up to a ton or more) were conducted and the extracts concentrated to obtain compounds that exist only in trace amounts but which are believed to be important flavor components. Over eighty volatile components of peas have been identified. Addition of some of these components singly and in combination to processed peas has not as yet resulted in improved flavor as judged by taste panels. In the course of this investigation, a new analytical procedure, called flash-exchange chromatography, was developed. It provides for the rapid analysis of trace amounts of volatile constituents and has wide applicability in the rapidly-advancing field of gas chromatography. The flavor of peas remains elusive and much research remains to resolve its composition and to establish the mechanisms of its development and its loss in processing.

3. Hop Oil Flavor Components. A fundamental study of the volatile flavor components of hop oil is supported, in part, by the United States Brewers Association, which provides the salaries of two chemists. The long-range purpose of this project is to isolate and characterize flavor components and to study their chemistry. Hop oil has been found to be exceedingly complex and it was necessary to fractionate whole oil into groups of components, similar in chemical structure, and further separate and characterize the compounds in

each group. It is estimated that approximately 200 volatile components exist in hop oil, some of which components exist in trace quantities. Beyond the basic complexity of the oil, it was found that hops from different sources vary greatly in their chemical makeup. Thus, European oils contain far less myrcene (a hydrocarbon) than domestic oils. That the varietal and geographic origins (possibly cultural practices, too) can so affect composition may provide the key to quality differences that exist between oils of different sources. Work has been initiated to catalog important compositional differences in hop oils, the same oils being evaluated by industry experts to seek a useful correlation that can be used to improve raw material for processing.

4. Nature of the Heat Resistance of Spores. The extreme resistance of bacterial spores to heat necessitates severe heat processing treatments, which in turn bring about flavor, color, texture, and nutritional deterioration in canned vegetables. A fundamental investigation of the nature of heat resistance exhibited by spores is expected to uncover facts that will make milder processes possible. In order to conduct this research it is necessary to have an abundant supply of appropriate bacterial spores, in a comparatively isolated state. Conditions were worked out for abundant sporulation of several bacterial species appropriate to the study, and a highly effective technique was developed to remove interfering vegetative cells and cellular debris from spore preparations. It is now possible to obtain good yields of clean spores from four species and to obtain smaller quantities of clean spores from natural sources. The recovery of clean spores from natural sources is especially important because food spoilage problems come from such. Cultured bacteria, induced to sporulate under laboratory conditions, while valuable for biochemical studies, may not have identical characteristics. Biochemical studies of spores and sporulation, conducted at the Western Regional Research Laboratory and, by contract, at the University of Illinois, provided observations demonstrating the association of heat resistance of spores to a specific acid component (dipicolinic acid) and to divalent metals (calcium, magnesium, and manganese). As spores form in bacteria, dipicolinic acid content increases. During the sporulation stage of development, calcium is essential to the increase in the acid content and the development of heat resistance. As spores are heated toward sterilization temperatures, a structural breakdown may occur, and the organism becomes susceptible to heat and is killed. Dipicolinic acid is released, as are the divalent metals. A heat shock, milder than that causing sterilization, will cause spores to germinate and become vegetative cells, which are heat sensitive. The great refractility of dormant spores has long been known and has been associated with an anhydrous state related to heat resistance. Findings of this research have contributed to a new concept concerning the maintenance of an anhydrous condition of spores. Many investigators have assumed the existence of a water-impermeable membrane.

Present knowledge of permeability properties of organic films suggests that an anhydrous state cannot be maintained by impermeability of so thin a membrane (spore dimensions will limit its maximum thickness). In addition, recent studies have indicated a free interchange with surrounding water of the small amount of water that exists in the spore. The new concept reconciles these contradictory observations and postulates the existence of a contractile layer in the spore which squeezes water out of the vital core and maintains it in a relatively anhydrous state by the exertion of external pressure. Preliminary evidence has been obtained in support of the new concept in the observation of a shrinking and swelling phenomenon, which occurs when spores in the early stages of germination are subjected alternately to mildly acidic and alkaline conditions. Evidence was obtained of complexing between dipicolinic acid and divalent metals in solutions and, similarly, in spores themselves.

5. Composition of Dry Beans as Related to Flatulence. An unpleasant physiological reaction (flatulence) is experienced by people who eat beans with the result that beans are not consumed in as great quantity as they might be. A basic study is being conducted, at the Western Regional Research Laboratory and, by contract, at the University of Illinois, of the causative chemical constituents and physiological and chemical mechanism concerned with bean-induced flatulence. Animal studies have been conducted in which, by feeding tests with beans and by injection of bean components into the upper intestines of anesthetized rats, irritation of the intestine walls, and intestinal swelling have been observed. Research is continuing to determine specific components in beans that cause these conditions. Feeding tests with human subjects provided quantitative measurement of flatulence under controlled dietary conditions and revealed that the species of bean (Lima vs. common), method of preparation (canned pork-and-beans vs. canned baked beans), degree of subdivision of the cell structure (homogenate of canned pork-and-beans vs. canned pork-and-beans), did not influence the amount of flatulence produced. The amount of beans in the diet did materially and consistently influence the quantity of flatulence (a 6- to 11-fold increase in intestinal gas with beans providing 27 percent of the calories in the diet compared to a control diet with none; a 13- to 16-fold increase, with 57 percent). The amount of carbon dioxide in the flatus increased linearly with the dry weight of the beans ingested, but there was no consistent relationsnip found with hydrogen or methane gas (two common components, along with carbon dioxide, of flatus). All three of these gases can originate from fermentation but carbon dioxide can result from other physiological sources such as the carbonates in digestive fluids and in the blood stream as a product of respiration. One postulate is that bean-induced flatulence is not of fermentative origin but related to the transfer of carbon dioxide through intestinal walls either by blocking transfer of that whose source is the

digestive secretions or by promoting that of respiration from the blood stream into the intestines. To pursue further the interesting leads developed in the contract studies at the University of Illinois, a new contract has been negotiated, funded by Administrator's contingency funds appropriated for 1962.

6. Composition of Dry Beans and Peas as Related to Cookability. A fundamental investigation of compositional factors that might relate to the cookability of dry beans and peas is conducted at Albany and Pasadena, California, supported, in part, by the California Lima Bean Advisory Board, which provides the salary of two chemists, and at the Fruit and Vegetable Canning and Quick-Freezing Research Association at Chipping-Campden, England, supported by a grant under P.L. 480. Internal reassignment of program strengthened dry bean research by the equivalent of 1.8 professional man-years, including 0.9 each transferred from research on other vegetables and deciduous fruit. Earlier published findings that the phosphorous-containing compound, phytate, was largely responsible for the degree of cookability of dry peas has not been substantiated by observation of this program. Research at Chipping-Campden has led to the conclusion that the texture of cooked dry beans is determined by the viscosity of the intercellular pectic material and the extent to which the tissue cells swell during cooking. The extensibility of the cell wall is held to be the dominant factor in determining the texture, with phytic acid concentration being of no significance. Contract research at the University of Idaho, now concluded, provided a comprehensive study to reveal possible correlations between selected compositional factors of a broad range of varieties of beans and peas and cookability. Of fourteen factors studied, only moisture content and alcohol-soluble nitrogen were significantly correlated with cooking time. At Pasadena and Albany, analytical methods are being developed and components of dry beans (including large Lima beans) are being catalogued for study of correlations that may exist with cookability. In part, these compositional studies are directed toward the changes that occur as beans mature. Subtle changes occur as beans mature and dry that materially affect the time required for cooking. These are not significant quantitative changes in the major constituents. Inositol phosphates (sometimes reported to be of importance in bean cookability because of their ability to complex with calcium) are being measured from beans of different maturities. A crystalline protein of beans has been isolated and is being characterized for physical properties and amino acid components.

7. Vegetable Pigments. The retention of natural color in processed vegetables is a major factor affecting the acceptability of products but it is rarely achieved. Fundamental research is conducted at the Western Regional Research Laboratory on chlorophyll and its measurement, and at the Low Temperature Research Station in Cambridge, England, under a P.L. 480 grant, on carotene and its volatile oxidation

products. A research contract at Brigham Young University on the measurement of chlorophyll and its breakdown products was concluded with the development of a new analytical procedure for chlorophylls a and b and pheophytins a and b. The method is considered more accurate than previous methods and its application to the measurement of chlorophyll retention and deterioration is in progress. Procedures for removing interfering substances from vegetable extracts are also being conducted. Carotenoid pigments in many green plants interfere with the spectrometric measurements of chlorophyll, and prevent a precise appraisal of chlorophyll deterioration. Separation methods have been developed that largely remove the interference and have improved the analysis of the important green pigments. The chemistry of oxidative and other types of deterioration of carotenoids that occur in the processing and storage of vegetable products is being studied. The relationship of carotenoid deterioration to specific off-flavors is being elucidated.

8. The Role of Sulfur Dioxide in Dehydrated Vegetables. A fundamental investigation of the chemical fate of sulfur dioxide or sulfite in dehydrated vegetables is being conducted at the Covent Garden Laboratory in London, England supported by a grant under P.L. 480, which is part of a project that includes research on potatoes, also. The objective of this research is to determine the chemical mechanism through which sulfite exerts its protective action on dehydrated vegetables. Model chemical systems have been used in which the effects of sulfur dioxide and other components have been observed as they affect simple browning reactants (e.g., glycine and glucose), and other more complicated chemical reactants (e.g., citral in place of glucose). By use of sulfur-35 as a radioactive tracer, the chemistry and migration of sulfite applied to potatoes during dehydration have been studied. By these means some of the complications of the mechanisms through which sulfur dioxide prevents non-enzymatic browning are beginning to unfold. A common chemical structure (the carbonyl group) in glucose and citral is involved in a reaction with amino acids, such as glycine, as a primary step in forming the brown pigment. Citral is more reactive than glycine because of its unsaturated structure. Sulfite seems to block browning by prior reaction with the carbonyl but unsaturation, as in citral, can be responsible for a migration of the sulfite within the molecule allowing a more rapid color formation by freeing the carbonyl. Effects of various reactants, e.g., calcium, iron, phosphate, and ascorbic acid (Vitamin C), on the rate of browning are being elucidated in this study.

9. Microbial Flora in Fruits and Vegetables. Fundamental studies on microbial flora within the tissues of fruits and vegetables have been conducted under P.L. 480 in the Department of Food Technology, Agricultural Research Station of the Ministry of Agriculture, Rehovot, Israel. Fruits have been found containing viable micro-organisms within the tissues. It is probable that these organisms gained entry

during the formation of the fruit on the plant, but this has not been conclusively demonstrated or proven. Species studied included tomatoes, cucumbers, green beans, broad beans, and peas. Bacteria of the Xanthomonas, Pseudomonas, Enterobacteria, and Corynebacteria groups have been found, as have yeasts of the Nematospora. Such entrapped micro-crganisms can make only a limited development and grow rapidly only when the tissue has been disturbed by injury or maceration. The relationship of such adventitious microbes to processing quality of fruits has not been revealed by these studies and may not yield itself to techniques that are currently available.

B. New and Improved Food Products

1. Tomato Powder. Using the new, foam-mat drying technique, tomato juice and tomato paste powders have been produced in the laboratory. Tomato powder has now been prepared commercially for a trial procurement by the Quartermaster Corps. Tomato juice can be made from foam-mat dried tomato powder having a natural, unburnt flavor, odor, color, and consistency. Flavor and color, however, are somewhat weak due to loss of volatile compounds during evaporation prior to drying and to an increase of the exposed surface during the dehydration process. Gas chromatographic analyses are being used to follow changes in volatile components in efforts to improve flavor of tomato powder.

2. Dry Bean Products. The slow-cooking property of dry beans and peas deters their utilization. A number of dry bean, pea, and lentil powders have been prepared by drum-drying slurries of cooked legumes, with or without seasoning. These products can be instantly reconstituted by addition of boiling water and used as soups, refried beans, and in various combination recipes such as meat loaf, party dips, etc. Excellent quality products have been produced and are receiving preliminary evaluations for marketing. The Idaho Bean Commission has been particularly active in demonstrating these products to interested food processors and in developing recipes for their use. Storage studies have indicated the moisture levels at which these products are stable enough for practical marketing. A low-moisture, nitrogen-pack has been found suitable. Added antioxidants extend storage life. The influence of moisture content of stored whole beans on quality of bean products is being investigated. In the higher moisture range (13 percent up to the spoilage level of about 17 percent) beans become significantly more difficult to cook after three to four months' storage. Edible chemical additives and enzyme preparations have been used in efforts to hasten soaking and cooking of dry beans. Some improvement has been accomplished with commercial preparations of cellulose splitting enzymes.

C. New and Improved Processing Technology

1. Foam-Mat Drying. Over 50 agricultural commodities, including tomato juice and tomato paste, have been successfully dehydrated by the foam-mat drying process invented by Department engineers at Albany, California. Continuous, automatic equipment has been designed and constructed for continuing experimental studies. A commercial dryer of similar design has been installed in a food processing plant in California and several other industry applications of this novel dehydration method are being investigated with pilot operations to develop commercial-scale equipment. Most products tested can be dried at atmospheric pressure without off-flavor development or discoloration. There is some loss of volatile flavor components. A major problem in the technology of each product so dried is to find means for adding back or redeveloping flavor. Laboratory tests were conducted in which volatiles released during concentration of tomato juice were trapped as a source of flavor components to be added back to powder to improve flavor. Plans are being drawn to collect such volatiles from commercial tomato processes, anticipating a fuller recovery of large quantities of volatiles from the initial heat treatments and concentration operations. Drying cycles for tomato products have been determined to minimize quality loss and to increase the heat and moisture transfer and mechanical efficiencies so that processing costs may be reduced.

2. Processing Quality of Vegetable Varieties. The selection of improved vegetable varieties with increased yield characteristics and insect- and pathogen-resistance is a never-completed task. With more and more vegetables going to markets in processed form, the processing characteristics of new selections must be given proper attention. At Puyallup, Washington, cooperative research is conducted on a continuing basis with the Washington State Experiment Station to evaluate processing quality of corn, beans, peas, and rhubarb relative to production conditions in the Pacific Northwest. In connection with these studies, maturity testing of corn relative to proper harvest for canning and freezing has been conducted. The puncture-pressure testing of sweet corn has been found to be of value as maturity increases during harvest season. However, season-to-season variation of absolute pressure values were encountered. A single maturity test for corn harvest is not available. Soluble solids and moisture content have shown a good correlation with maturity under some conditions but the relationship can be upset by rain, which may increase moisture levels and obscure the maturation measurement.

3. Freezing and Dehydrofreezing. Limited investigations on freezing and dehydrofreezing vegetables are carried out to determine stability of the various product quality factors during processing and subsequent storage as they are affected by processing methods used. Studies of the green color retention of Brussels sprouts relative to

blanching method have indicated that a preliminary warming of the product at 130° F., prior to steam blanching, allows a higher retention of chlorophyll. Preheating above 150° causes a greater loss than in controls not preheated. Chlorophyll retention studies of stored dehydrofrozen peas relative to conditions of dehydration are in progress.

4. Microbiology of Frozen Vegetables. Maximum tolerances for viable microbes in frozen vegetables are specified by some commercial buyers. Analytical techniques to measure microbial contamination in processing plants as a guide for operations control are being developed. Their application to industrial practice is studied by informal cooperation with frozen food processors in the Pacific Northwest by the staff of the field station at Puyallup, Washington. Work has been conducted in three plants processing peas and 15 plants processing corn. Focal points of contamination have been found on conveyor belts and in hoppers, froth-flotation cleaners, dewatering shakers, and improperly built flumes and other spots where entrainment of the product may occur. The analytical methods used have shown good possibilities for control of the practices that lead to unusual build-up of microbial contamination. The conditions vary so greatly from plant to plant that continuation of this work is important to be certain that the applicability of the methods developed is universal.

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Area 9 CASTOR, SAFFLOWER AND OTHER WESTERN OILSEEDS--PROCESSING AND PRODUCTS

Problem. To provide valuable diversification crops for the acreage withdrawn from the production of cotton, wheat, feed grains, and other surplus crops, there is a critical need to expand the markets for crops such as castor and safflower. But these crops are so new to our agricultural economy that their market potential has not been adequately developed. Castor and safflower have good potential because of the unusual properties of their oils. The possibility of large-scale increases in the production of these oilseeds would be strengthened if high-quality feed products could be developed from the oilseed meals. Basic information is needed on the composition of the oils and of the meals left after extraction of the oil, and this, in turn, requires the development of adequate analytical methodology. Rapid and accurate analytical methods are needed to control and improve the processing of the oils and meals for food, feed and industrial applications. Research on chemical conversion of the oils and evaluation of the modified products is needed to find new or improved large-volume uses. The high percentage of linoleic acid (essential fatty acid) in safflower oil points to a rapidly expanding usage as a food oil. But this same fatty acid imparts a high degree of susceptibility to autoxidation. Research is needed to stabilize safflower oil in various types of food products. Improved procedures for decorticating and processing castor and safflower seeds are needed. There is a particularly critical need to develop methods for the removal or destruction of the allergenic and toxic components of castor meal which presently limit its use to fertilizer. Research to isolate and characterize the constituents in castor and safflower meals is needed to develop non-toxic, non-allergenic feed and food products of high value. Basic and applied research is needed to prepare chemically modified products from the meals for industrial applications, to develop economical procedures for carrying out the modifications, and to evaluate the modified products.

USDA PROGRAM

In the Western Utilization Research and Development Division, both basic and applied research are conducted on castor seed at the Division headquarters at Albany, California and, under contract, at Tucson, Arizona and Menlo Park, California. Basic, compositional studies on castor seed meal are concerned with the resolution of its water-soluble proteins and determination of the allergenic and antigenic properties of these components. Studies are conducted on the composition of castor oil, and new analytical techniques are developed.

Applied research on castor meal has as its objective the development of economical methods for deallergenizing the meal without impairing its nutritive quality, to increase its value as an animal feed ingredient. Castor oil and its major constituent, ricinoleic acid, are being studied to provide for them new and improved industrial applications. Thus, methods are being developed for the preparation of various types of polyurethane foams incorporating castor oil or its derivatives. Procedures are also being devised for the preparation of chemical derivatives of ricinoleic acid, including a number of amides and phosphate esters. Several of the latter compounds may be useful for improving the flame-resistant properties of castor-based polyurethane foams of the type which may be used for building insulation. The utility of various polymerizable monomers, derivable from castor oil, for the production of synthetic polymers for use in rubbers, plastics, etc., is being investigated under contract.

The Federal program of research in this area totals 11.5 professional man-years. Of this total, 4.8 are assigned to chemical composition and physical properties; and 6.7 to new and improved products.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 0.4 professional man-years of research on chemical composition and physical properties. This research is designed to evaluate the practicability of producing animal feed from castor pomace, by removing toxins and allergens. This program, which is cooperative with the USDA and industry, also includes studies of the biosynthesis of the alkaloid, ricinine, and the amino acid content and biological value of castor seed protein.

Industry and other organizations are estimated to be conducting research on castor with expenditures equivalent to about 14 professional man-years. Most of this effort is directed, by oilseed processing and chemical companies, toward the development of new industrial uses for castor oil and its derivatives. Only negligible effort is being expended on basic research on castor, either by industry or academic institutions. About 10 professional man-years of effort are being applied by industry to research on safflower. Until very recently, most of this research was devoted to developing increased industrial and feed outlets for safflower oil and meal. Increased effort is now being directed toward the utilization of the oil in edible products where it is valued for its high content of linoleic acid.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Allergenic and Antigenic Proteins. Under a contract project, concluded during this reporting period, information was obtained on the preparation and chemical properties of the highly allergenic protein complex of castor seed meal. A quantity of this material, produced by the contractor, was fractionated and characterized by several techniques. Each of these antigenic fractions was shown to be an extremely potent allergen for humans sensitive to castor. Deallergenization of castor meal will, therefore, require the inactivation of all of the antigenic materials present. Treatment of castor meal to convert these protein materials into innocuous compounds would be expected to affect other proteins present and thus lower the nutritive value of the treated meal. More detailed information regarding the chemical composition of the antigenic proteins could possibly lead to the development of selective inactivation procedures to avoid destruction of nutritionally valuable proteins. Studies on the isolation and characterization of the individual antigens of castor are in progress, under contract, at the Stanford Research Institute. Although hanging curtain electrophoresis can be used to resolve a partially purified allergen complex into a number of discrete fractions, the technique fails in use with the crude allergen mixture. For this reason, methods are being examined for accomplishing a subfractionation of the crude mixture preparatory to electrophoretic separation of discrete antigens. Confirmation has been obtained of preliminary findings that castor pollen, female flower blossoms, and seed proteins cross react. Thus, castor pollen can sensitize an individual to the meal (and vice versa). This may explain the puzzling cases of individuals with no history of prior exposure to castor meal showing symptoms of allergy when exposed for the first time. It was also shown that castor-allergic individuals react to, and may have been sensitized by, other members of the family Euphorbiaceae (spurges). It appears that allergy to castor meal is probably a complex of allergies traceable to both the castor antigens, and the antigens that may be present in a wide variety of contaminating materials, especially when castor is harvested mechanically.

2. Detection of Allergenicity and Antigenicity. The study of the composition of the individual antigenic proteins of castor requires a reliable allergy assay technique. Such an assay is also necessary for evaluating the effectiveness of methods being tested for deallergenizing castor meal. It was shown early in this work that the commonly-used Schultz-Dale *in vitro* assay for allergenicity was of little value because of its lack of specificity. In addition to the truly antigenic proteins of castor, a factor is also present which produces a non-specific, histamine-like response resulting in a positive Schultz-Dale test even in the absence of a true allergenic

reaction. Testing allergens or antigens on humans is a highly undesirable procedure because of the risk of allergic shock or hepatitis infection. For these reasons an animal assay technique has been developed which has been shown to be accurate, specific, rapid and relatively inexpensive. This technique depends upon the phenomenon of passive cutaneous anaphylaxis (P.C.A.). It is this phenomenon which causes a local sensitization in the skin of a non-allergic individual following the injection of blood serum from an allergic individual. The sensitized skin sites will often exhibit an inflammatory reaction with wheal formation, upon direct injection into the site of a minute quantity of the substance (antigen) causing allergy in the serum donor. The inflamed area of the skin can be made highly visible by the intravenous injection of a colloidal dye, such as Evans Blue, prior to challenging with the antigen. Because the minute blood vessels in the inflamed area exhibit increased permeability, dye leaks into the extravascular spaces in the skin and a blue spot appears, indicative of a positive allergy test. This technique was developed using rabbit antisera in the skins of guinea pigs but has since been extended to two species of monkeys. Results have indicated an essentially perfect direct correlation of the P.C.A. tests with observations on humans. The validity of the monkey P.C.A. test has been demonstrated with known and probable contaminants of castor meal. It was successfully applied to the detection of allergies due to castor plant pollen, ragweed, animal danders, cereal grains, grass pollen, and insects commonly associated with castor beans. This technique will, therefore, enable investigators to differentiate between true castor allergy and reactions to contaminating components of castor meal.

3. Fatty Acids of Castor Oil. New and improved methods have been developed for the analysis of mixtures of fatty acids or their derivatives. The thin layer chromatostrip technique has been adapted and used effectively in combination with suitable detection systems for identifying compounds derived from castor oil and for following the course of reactions, monitoring purifications, determining purity and for isolating reaction products. In addition, a silicic acid column liquid partition chromatographic method has been adapted to the separation and analysis of the nonhydroxy, monohydroxy, and dihydroxy acids and methyl esters obtained from castor oil. This procedure, in combination with gas-liquid chromatography of the separated esters, should make possible relatively simple yet accurate determination of the fatty acid composition of castor oils.

B. New and Improved Products

1. Chemical Derivatives. The preparation of amides of hydroxy acids such as ricinoleic acid has been greatly facilitated by the development of a simple, rapid and versatile method of synthesis. Excellent yields (80% or higher) of amides are obtained from mixed carboxylic-carbonic anhydrides and amines. Unlike other methods of activation

of the carboxyl group, the mixed anhydride method does not appreciably affect the hydroxyl function of the hydroxy acids studied. In addition, the mixed anhydrides from castor-derived acids appear quite insensitive to steric hindrance in the amines used. Amides have been prepared using mixed anhydrides from the castor-derived ricinelaidic, 12-hydroxystearic, dihydroxy- and trihydroxystearic acids. Thus far, over twenty new amides have been prepared by this method of synthesis and are being characterized. Several amides are being evaluated, industrially, for use as mold release agents, components of water repellent formulations, etc. Studies are in progress, under contract, to evaluate castor oil-derived monomers for the production of copolymers suitable for industrial use in plastics, etc. Vinyl 12-hydroxystearate has been prepared and its polymerization is being studied. During the preparation of this compound an intermediate was obtained which appears to be an addition compound of vinyl acetate and vinyl hydroxyoleate. This addition compound is readily hydrolyzable to vinyl 12-hydroxystearate or 12-hydroxystearic acid. The contractor has shown that vinyl 12-hydroxystearate may be polymerized in hexane solution using azo-bisisobutyronitrile to initiate polymerization. The polymer was obtained in about 45% yield and was a colorless solid, soluble in tetrahydrofuran, with an inherent viscosity of 0.11 and a softening range of 65 to 75° C. The intermediate (addition compound) polymerized in benzene solution with the same nitrile initiating compound and was converted quantitatively to a polymer which proved to be a tetrahydrofuran-insoluble gel. These studies are being continued and will be extended to other castor oil-derived monomers. Castor oil and certain of its derivatives were tested in rats to elucidate the specific structural configuration responsible for cathartic activity. Results indicate that both the hydroxyl function on carbon 12, and the double bond between carbons 9 and 10 of the ricinoleic acid moiety are essential for cathartic action; this activity is lost upon either masking of the hydroxyl group or hydrogenation of the double bond.

2. Urethane Foams. It has been demonstrated that low-cost polyhydroxy mixtures based on castor oil can form inexpensive, solvent-blown urethane foams. Conditions necessary for the production of strong, uniform, low-density, rigid polyurethane foams using a halomethane (CCl_3F) as a blowing agent were investigated with special emphasis on the use of low-cost castor oil-polyol mixtures (rather than more expensive castor-based derivatives). Castor oil cannot be used alone to make rigid, solvent-blown foams. Excessive shrinkage occurs because of a relatively low ratio of hydroxyl content to molecular weight. One remedy, which was investigated previously, is to modify the castor oil to add more hydroxyl groups, but the process is expensive. An alternate approach, one that keeps the cost down, is to blend the castor oil with inexpensive lower molecular weight polyols to bring the hydroxy-to-weight ratio up. This was done by using solutions of triisopropanolamine and of mixtures of triisopropanolamine and triethanolamine in castor oil as the polyol

components. Foams were prepared by reacting these polyol mixtures, in the presence of catalyst, surfactant and CCl_3F with prepolymers prepared from toluene diisocyanate and certain polyether polyols or mixtures of these polyether polyols with castor oil. The effect of polyol and prepolymer composition, and blowing agent concentration on such foam properties as density and compressive strength was investigated. It was found that compressive strengths are more closely related to urethane group concentration than to cross link site concentration. The properties of castor oil-based foams were also compared with those of the competitive polyether-based foams. A series of foams was prepared in which the polyol portion was varied so that in one case it consisted entirely of a commercial polyether (equiv. wt. 101); in a second case it was a castor oil-triisopropanolamine mixture (equiv. wt. 100); and the third contained an equal mixture of the two polyol types. The castor oil-based foams were slightly stronger and had better dimensional stability after humid aging, but exhibited slightly higher initial shrinkage. The thermal conductivity of the castor oil-based foams increased more rapidly on aging than did the polyether-based foams. Reasons for this phenomenon are being sought, as are means for improving the fire-retardancy of the urethane foams. Such foams can be made self-extinguishing by incorporating, into their formulations, 10 to 20% of certain phosphorous- and halogen-containing compounds. In general, the properties of castor oil-based urethane foams are comparable to those of foams obtained from more costly polyols. In a highly competitive market, these lower cost urethane foam formulations have stimulated considerable industrial interest.

3. Animal Feed Meal. Deallergenized castor meal would constitute a suitable ingredient for livestock and poultry feeds and would, as such, command a considerably higher price than it could as a general purpose agricultural fertilizer. Several methods for deallergenizing the meal are being investigated, including variations of the alkali cooking procedures first used at the Southern Utilization Research and Development Division, New Orleans, Louisiana. The effectiveness of deallergenization treatments is determined most accurately by means of the P.C.A. test (described earlier). Treatment for inactivation of the allergens may be attempted at one or more of several stages of the processing of castor. The most promising stage appears to be immediately following desolventization of the extracted pomace. Preliminary studies indicate that the hull content, moisture level, and particle size of the material all affect the efficiency of treatment. Sufficient treating agent must be used to insure that all of the meal is exposed, but excess moisture will increase drying costs, thus these factors must be balanced carefully. Laboratory-scale treatments tested include: alkaline hydrolysis (sodium, potassium, ammonium, and calcium hydroxides), oxidation, and ammoniation (gaseous). Several such treated samples, notably those treated with sodium and

potassium hydroxide, appeared to have been nearly completely deallergenized. Furthermore, preliminary feeding trials using chicks, indicated that the nutritive value of these treated meals had not been impaired. A series of experiments was conducted to test the value of castor meal as a soybean meal replacement in a chick ration. It was found that, when lysine was added, castor meal could satisfactorily replace 50% of the soybean meal in the ration. At this degree of substitution, the actual level of castor meal in the ration was almost 25%. These experiments offer considerable promise for the development of practical methods for deallergenizing castor meal. However, the laboratory-scale experiments conducted to date will require much modification before economically feasible large-scale processing procedures may be recommended.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

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New and Improved Products

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Area 10 SUGAR BEETS-- PROCESSING AND PRODUCTS

Problem. Sugar beets are grown mainly to be processed for the manufacture of sugar, while a very small proportion is used for livestock feed. The most important processing problem facing the sugar beet industry, which affects both growers and processors, is the recent marked decline in sugar beet quality. In addition to a decline in average sucrose content of beets, the recovery of sugar in processing has declined because of increases in soluble non-sugar impurities. The traditional processing methods for sugar manufacture cannot cope with these beets whose lower quality is due in part to excess nitrogen fertilizer, used to improve tonnage yields. Improved processing procedures should benefit both the growers and processors. It is known that small concentrations of certain chemicals in beets affect processing quality but not enough information is yet available to devise new economical processing procedures that are required for efficient recovery of sugar from high-impurity beets. Because costs of producing beets and processing sugar are rising and consumption and price of sugar are essentially constant, all factors important in utilizing the crop must be continually examined with the objective of improving processes. There is still a lack of knowledge of the composition of sugar beets, juices, pulp, and crude sugar to achieve this objective, and sugar losses resulting from spoilage and respiration of beets held at processing plants cannot be prevented by existing methods. Only an expanded research program can provide the needed information at an early date.

USDA PROGRAM

Both basic research and process development studies on sugar beets are being conducted in the Western Utilization Research and Development Division's headquarters laboratory at Albany, California, under contract at Fort Collins, Colorado and, under a Public Law 480 grant, in London, England. The basic research program involves a comprehensive study of the naturally-occurring sugar beet and beet juice constituents, both carbohydrate and non-carbohydrate. Biochemical studies of the carbohydrate constituents seek to determine the origin of their formation, leading to the development of methods for the reduction or elimination of those which lead to decreased yields of sucrose. Chemical studies of the non-carbohydrate constituents provide information needed for the development of methods for negating or ameliorating the adverse affects on processing which are exerted by many of these constituents. Contract research, under multiple sponsorship, is providing information on the variability of specific

non-sugar chemical constituents in beets of known genetic and agronomic history. Although Federal research on new products from sucrose (sucrochemicals) has been terminated, studies continue in England, under a P.L. 480 grant, on the reactions of sucrose with constituents of vegetable and animal fats and oils, to produce new and useful compounds having special hydrophilic and lipophilic properties. Processing research on sugar beets deals with the effects of the many variables which influence the efficiency of recovery of sucrose. Pilot-scale sugar beet processing facilities are used to test these processing variables and to evaluate new and improved processing techniques.

The Federal program of research in this area totals 6.6 professional man-years. Of this total, 4.2 are assigned to chemical composition and physical properties; 0.0 to new and improved products; and 2.4 to new and improved processing technology. In addition the Division sponsors, under P.L. 480, 3.3 professional man-years of research on new and improved products.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported no research in this area.

Industry and other organizations are estimated to be conducting research on sugar beets (or on sugar, applicable to beet sugar) with expenditures equivalent to about 26 professional man-years. Almost one-half of this effort is expended by beet sugar companies to improve processing efficiency. The remaining effort, that of chemical manufacturing companies and the Sugar Research Foundation, Inc., is being applied to the development of new uses for sugar, especially as sucrochemicals for non-food purposes.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Carbohydrate Constituents. Probably the major deleterious carbohydrate in beet juices is the trisaccharide, raffinose (a combination of sucrose and galactose). Raffinose interferes with normal sucrose crystallization by both slowing its rate and also causing very elongated crystals to form. It has been shown that holding beets at low temperatures prior to processing (common in the Rocky Mountain states), causes raffinose to be produced in quantities which seriously hamper processing operations. After 3 months at 34°F. raffinose contents were found to have increased from 0.25 to 1.25% based on sucrose. At this level, sucrose crystallization time is more than doubled, and crystals may be so extremely elongated as to make it impossible to pack 100 pounds of sugar into a standard bag. Raffinose

levels of 3 to 4% are sometimes encountered, resulting in a shutting down of the crystal end of some factories and the production of more molasses. Studies in progress are designed to determine the critical temperature for raffinose accumulation so that processors would know when it is necessary to speed processing to minimize sugar losses. Also under investigation are means for reducing the raffinose content of beets and juices by: a) enzymatic (alpha-galactosidase) conversion of raffinose to sucrose and galactose; b) conversion of raffinose to a reducing trisaccharide by the action of galactose oxidase; and, c) chemical inhibition of raffinose biosynthesis. Of prime importance in all of these studies is an accurate and sensitive analytical method for following changes in the raffinose contents of beet tissues and juices. Existing chemical and paper chromatographic assay methods are inadequate for this purpose and two biochemical methods have been developed which show much promise. These methods are being refined and evaluated. Basic investigations were continued on the rate and mechanism of sucrose crystallization. An apparatus designed to measure surface areas of powders (such as powdered sugar) by continuous gas flow adsorption, was used to obtain surface area data required for interpretation of observations of sucrose crystallization using a multiple seed technique. Some modification of the apparatus was necessary before acceptable surface area measurements were possible. Further improvements will be required before this technique will be completely satisfactory. The crystallization of sucrose complexes was studied as a means for obtaining higher recovery of sucrose from molasses. Sucrose calcium chloride hydrate was successfully crystallized from aqueous solution and optical and crystallographic studies were made on these unique crystals. This technique will be pursued further.

2. Non-Carbohydrate Constituents. Statistical evaluation of data from analyses of beet molasses over a period of several years show a significant positive correlation of chloride content with molasses purity. The higher the chloride content of molasses, the higher the sugar content of the molasses and, therefore, the greater the sugar loss during processing. Because chloride analyses have been laborious to perform, such data were not usually obtained during chemical control in sugar manufacturing. A commercial machine capable of analyzing juices for chloride recently became available and, in an attempt to verify and extend these laboratory findings, a cooperative study was arranged with a large sugar beet processor. Samples were obtained from a factory having high apparent sugar loss and high molasses chloride content, and analyses were performed using this newly-developed automatic chloride titrator. It was found that: a) the main source of chloride entering beet factory juice is the beets themselves; b) chloride passes through the beet juice purification process into the sugar juices and accumulates in the molasses; c) it appears possible to predict the average true purity of molasses

to be produced, from the purity and chloride content of the beet thin juice; and d) the relationship of molasses chloride to molasses purity was confirmed and agreed with data previously obtained in laboratory studies. Calculations showed that a pound of chloride carries 4 to 6 pounds of sugar into molasses and that removal of chloride from molasses would increase sugar recovery by 80 to 120 pounds per ton of molasses. Extensive analyses were completed of samples of molasses from 18 straight house and 12 Steffen house sugar beet factories. In addition to the melassigenic effect of chloride, it was found that all nitrogen fractions except nitrate have a significant negative correlation with purity, indicating that these compounds are not as deleterious as has been assumed by many in the industry. Contract research continues on the effect of genetic and agronomic factors on non-sugar constituents of sugar beets. Preliminary data show definite interactions between genotypes and environments, and the existence of favorable phenotypic--dominance phenomena. The F_1 hybrid beet, grown and studied under this contract, was shown to contain much less chloride under all fertility levels than other beets tested. Considerably higher sugar recovery should be possible from this beet variety since loss of sugar in molasses is markedly reduced when less chloride is present. Once sufficient information is obtained it would appear possible to produce sugar beets having lower levels of non-sugar constituents, e.g., chloride, nitrogen compounds, etc., by proper control of variety, cultural, and fertilization practices. Most samples of raw beet sugar contain one or more objectionable odorous amine-like chemicals which occasionally persist through melting and recrystallization and are detected in the refined sugar. Conventional methods having been unsuccessful in identifying these materials, hydrogen flame ionization gas chromatography was applied. Analysis of a sample of air from above raw beet sugar showed that at low temperature (24° C.) one component occurs in the parts per trillion range. A high temperature analysis failed to disclose any detectable volatile constituents. These results indicate that the odoriferous materials are very high boiling and occur in exceedingly low concentrations. Further attempts will be made to identify these beet sugar constituents.

3. Sugar Sorghum Composition. Samples were obtained of the 1961 crop of Brawley variety sweet sorghum, developed by personnel of the Crops Research Division. Preliminary evaluation of 1960 samples had shown a higher than expected invert sugar content, but an indication that invert disappeared during cold storage was not confirmed with the 1961 samples. Analysis of field-stored canes showed that reducing sugar was much higher than was expected by Crops Research Division, and that polarimetry does not give the correct sucrose value. Paper chromatographic analysis showed that, although total glucose plus fructose is the same as that found by chemical assay of reducing sugar, glucose concentration is about three times that of fructose. This explains, in part, the observation that polarization values are too high, and also may explain the previously low invert sugar concentrations found in

Brawley sorghum by polarization methods. Because of the excessive content of color and lime salts, and relatively low purity, compared to sugar beet juices, it appears that development of sugar sorghum as a complementary source of crystalline sucrose will require a very low invert variety or adaptation of modified cane sugar processing techniques.

B. New and Improved Products

1. Sucrochemicals. The Federal program of research in sucrochemistry has been terminated. Prior to termination of these studies, however, considerable information was obtained on the relative reactivities of the hydroxyl groups of the sucrose molecule, and silicic acid and gas chromatographic techniques were adapted for the analysis of sucrose derivatives. Several such derivatives were prepared for evaluation as surfactants, etc. Improved methods were developed for methylating sucrose (and other sugars), and both silicic acid and gas chromatography were shown to be extremely useful for the separation of these methylated sugars as well as for other substituted sugars. A combination of high resolution separation, collection, crystallization, and X-ray diffraction permitted identification of the compounds. It was determined, for example, that in sucrose monostearate the major sites of substitution are on the glucose moiety of the sucrose molecule, particularly carbon-6 of glucose. Silicic acid "chromatostrip" chromatography was used successfully to separate such sugar mixtures as sucrose and raffinose, and mono-, di- and tri-fatty acid esters of sucrose. Information gained from these studies, and the analytical techniques perfected, are expected to be of considerable value in research on the nature and occurrence of certain known, or as yet unidentified, non-sucrose sugars in sugar beets and process liquors. Research on sucrochemicals is still proceeding under a P.L. 480 grant executed in 1959. This work is aimed at synthesizing sucrose fatty esters with appreciable surface activity and good solubility in water. Such compounds are highly desirable for use as detergents and emulsifiers. Sucrose mono-12-hydroxy-stearate was prepared and, although water-soluble, was found to be insufficiently surface-active. Attempts were made to increase the water-solubility of sucrose stearate by the preparation of the phosphate, sulfate, etc., derivatives, and also by mild oxidation of the stearate ester, but these attempts proved to be unsuccessful. The preparation of other types of sucrose esters, such as those with tartaric and lactic acids, is being studied.

C. New and Improved Processing Technology

1. Juice Diffusion. Sugar beet processors have been perplexed by the odd distribution pattern of lactic acid produced by fermentation in continuous countercurrent diffusion batteries. This phenomenon was explained several years ago in this laboratory by theoretical calculations on the operation of continuous countercurrent sugar beet

diffusion batteries. The simplifying assumptions were that equilibrium is attained between juice and pulp-liquid in each cell; that transfer consists of moving all of the juice forward and all of the pulp and pulp-liquid backward; and that there is no selective membrane effect in the pulp-liquid phase. From these theoretical considerations it appeared that an appreciable fraction of soluble solids in battery supply water would be extracted by the pulp leaving the battery and thus would not appear in the juice. This is important because diffusion battery supply water used in extraction of sugar from beets often contains soluble impurities such as salts that cause losses of sugar through the formation of additional molasses. Practical tests were made with a laboratory continuous countercurrent diffuser using 5% salt water as the battery supply. At equilibrium, only 52% of the salt was discharged with the pulp although its salt concentration reached 5%. Forty-eight percent of the added salt was carried forward with the juice due to the high ratio of water to pulp at the tail end of the battery. Decreasing the ratio of juice to pulp would enable more salt to be removed by pulp-liquid but at the same time would decrease sugar extraction efficiency. Alternatively it should be possible to attain efficient sugar extraction and at the same time remove salt from the battery supply if the battery supply could be split into two streams, only one of which contained salts. In beet sugar factories two sources of battery supply water are available. One source comes from wells or municipal supply and often contains salts, and the other is distilled water as condensate from the evaporation of sugar liquors. The use of the salt water at the tail end and the distilled water somewhere toward the head end of the diffusion battery should give the desired effect of a low juice to pulp ratio at the tail end where salt is diffusing into the pulp-liquid and the high ratio of juice to pulp-liquid toward the head end where sugar is diffusing out of the pulp-liquid. The results of the practical test in the laboratory diffuser with salt water and distilled water used as split-stream battery supply showed 87% of the salt was eliminated in the pulp-liquid and only 13% into the juice. Sugar extraction efficiency was almost unaffected. These results are very important and show that with proper manipulation of a split-stream battery water supply, immediate efficiencies in diffusion of sugar from beets could be realized with no increases in processing costs.

2. Juice Purification. An extensive comparison of the effects of alkalinity and pH during first carbonation purification of juices from beets grown in, and representative of, most of the major production areas of the U.S., established that pH is the critical factor controlling the efficiency and capacity of the present-day purification process. Continuous pH control in these experiments was extremely difficult because of fouling of the glass electrode assembly from some juice constituent. Tests were made using a new type antimony pH electrode which had been reported to be less subject to fouling. Under the conditions of this experiment where pH values of 10.15 at 80° C. were

measured, the antimony electrode offered no advantage over the use of the glass electrode because it was difficult to standardize as to an exact pH value, it was hypersensitive to changes of temperature, and the steady drift of pH indicated that some continuous change in its surface occurred. It was shown that sedimentation rates were negatively correlated with pH. Furthermore, it was found that all juices studied could be optimally processed at pH 10.1 to 10.2, measured at 80° C. Due to great differences in buffering capacity between different lots of beets, there was a wide range of alkalinities corresponding to the optimum pH value. Use of this information will assist beet processors to maintain satisfactory operation rates on beets of varying character, a problem which has always existed in the past since the settling capacity of the factory is sometimes the factor limiting the feed rate of the raw material.

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Area 11 POULTRY-- PROCESSING AND PRODUCTS

Problem. The $1.6 billion poultry meat industry is now operating on very narrow or negative profit margins. This industry is confronted with the problem of converting continually increasing amounts of poultry into a wide variety of products having high quality and improved convenience, at costs attractive to consumers and remunerative to the poultry grower. Information on the properties and processing characteristics of poultry is not sufficient to enable us to better utilize poultry in a variety of forms attractive to consumers. Increased utilization of poultry would also serve toward eliminating our feed grain surplus, increasing returns to farmers and providing better products for American consumers.

Although poultry is a highly efficient converter of feed to meat, more grain is used by poultry per calorie of food produced than by any other commercial animal because a high percentage of the poultry diet is grain and because poultry meat contains exceedingly little fat. Furthermore, one-fourth of all grain fed to animals is used for poultry and egg production. For these reasons, increased consumption of poultry products would be a highly effective means of providing increased markets for surplus grain. Also, the efficiency of feed utilization by poultry makes possible prices of poultry low enough to be within reach of increasing numbers of consumers. A still further benefit would arise from the increased use of poultry by improving the nutrition of consumers having diets now low in animal protein.

The consumption of poultry has steadily increased from 23 to 28 to 36 lbs. per capita for 1949, 1954, and 1959. This important increase has involved the factors: price, quality of product, availability, and disposable income. Because of the current low profit margin it is impractical to increase consumption by lowering farm prices. Increased demand for and consumption of poultry will require higher quality and more convenient products and a greater variety of them to meet the desires of the modern consumer. However, in addition to greater returns from increased demand, a greater profit margin for the farmer can, of course, come from greater efficiencies in processing.

The trend toward convenience foods and further processing has primarily involved the development of precooked poultry products, which are generally less stable, are subject to warmed-over flavors, and are more likely to provide texture problems than uncooked items. With the expansion in scale of operation and the emphasis on continuous, more efficient processing, need has arisen for improved

processing procedures for feather removal, chilling, tenderization, freezing, deboning, and commercial cooking. Lowering the cost and improving the quality of products that can be stored at ambient temperatures, such as canned, dried, cured, and irradiated products, offer further potential for poultry utilization in domestic and export markets. As a foundation for applied studies, further knowledge is needed on the chemical nature of flavor and flavor changes in processing and storage, on tenderness development, and on proteins, lipids, and other components in relation to processing and new products.

USDA PROGRAM

Basic and applied research on poultry meat and poultry meat products are conducted at the Division headquarters at Albany, California and, by contract, in East Lansing, Michigan. Fundamental studies on poultry flavor are concerned with the identification of flavor precursor constituents in poultry meat and in the isolation and identification of volatile flavor components developed during the cooking of poultry. The chemistry of muscle protein and post mortem chemical changes are investigated relative to the tenderness and other quality characteristics of poultry. The basic physiological character of feather release mechanism in fowls is studied to provide a foundation for improved feather removal. Applied research is conducted on the stability of cold-tolerant organisms; special problems of flavor, texture, and stability of precooked frozen foods; processing factors that influence tenderness of poultry meat; and, supported by transfer of funds from the Department of Defense Quartermaster Research and Development Command, preservation of poultry meat by use of ionizing radiation.

The Federal program of research in this area totals 13.9 professional man-years. Of this number, 7.6 are assigned to chemical composition and physical properties; 4.3 to new and improved food products (including 1 supported by transferred funds from the Defense Department); and 2.0 to new and improved processing technology.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported 14.5 professional man-years divided among subheadings as follows: chemical composition and physical properties 3.2, new and improved products 5.5; new and improved processing technology 5.6; and new and improved uses for feathers and other processing wastes 0.2. Fundamental studies are concerned with the effects of dietary factors on poultry meat quality, physico-chemical measures of changes in quality, and physico-chemical changes in poultry meat during post mortem period. Applied research is concerned with development of new frozen and otherwise

preserved poultry meat products and suitable outlets for less desired, bony parts. Processing studies include factors to reduce quality defects, shrink, and nutrient loss; improved freezing methods; and use of anesthetizing agents and their effects on blood loss, feather removal, and tenderness. A small amount of work is conducted on finding new and improved uses for feathers and other poultry by-products.

Industry and other organizations including processing companies, and equipment and packaging manufacturers conduct research programs that are principally concerned with specific applications to individual corporate problems. Such investigations are connected with the development of processing and raw material control methods, more efficient handling and processing procedures, prevention of quality defects and deterioration, new product formulations, and development of processing equipment and protective packages. The findings of such research is frequently kept confidential or protected by patents. The fundamental requirements of processing and packaging are developed in State and Federal laboratories and the development of equipment and packages to meet the requirements accomplished by industry research. Universities and non-profit research institutes conduct poultry research on a limited scale, often with contract or grant funds from public resources. Estimated annual expenditures in this area are equivalent to approximately 60 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Flavor Chemistry. Fundamental investigations on the chemistry of poultry flavor are conducted to provide information that can guide a rational approach to preservation of optimum quality of poultry meat products. Volatile components of cooked chicken and storage deteriorated chicken products are separated and characterized by gas chromatography. Correlations are sought with sensory evaluations. Decadienal has been identified as a principal volatile component, arising from the fatty portion of chicken meat during cooking. Synthetic decadienal, on exposure to air develops a stale and then a rancid odor. It is thus suspect as a precursor of these types of off-flavor. Rancidity developed in stored, frozen fried chicken was detected by organoleptic evaluations sooner than it could be detected by the gas chromatographic equipment available. As storage continued (8 and 10 months at 10° F.) chromatograms showed a very definite increase in amount and complexity of volatiles formed. More sensitive equipment is being constructed. Volatile carbonyls of cooked chicken have been isolated and characterized in previous studies. It has now been found that carbonyl compounds tend to accumulate in the fatty phase of the broth where their contribution to off-flavor is obscured.

Isolated carbonyls added back to the aqueous portion of the broth cause easily-detected off-odor. Broth flavor was adversely affected when acidity was decreased either by ante mortem injection of adrenalin or by cooking immediately post mortem. Increased pH resulting from both methods may be influencing flavor producing reactions during cooking.

Studies on the precursors of hydrogen sulfide, which is continuously evolved during the cooking of chicken, have indicated that it comes mainly from the proteins and only in minor proportion from small molecular weight sulfhydryl compounds such as cystine or glutathione.

2. Effects of Production Factors on Poultry Flavor. Studies were conducted cooperatively with the Poultry Research Branch, Animal Husbandry Research Division, to determine differences in chicken flavor that might exist because of breed, feed, age, and sex of bird. Comparisons were made at age differences ranging from 6 to 78 weeks, and using several different cooking methods. In general, flavor differences were found to be small and probably of negligible practical importance. Since consistent, large differences were not found, it does not seem necessary to replicate such research with a range of age in the products under investigation.

3. Post Mortem Biochemistry and Tenderness. Fundamental research is conducted on the chemistry of protein change in poultry flesh following slaughter and the effect of chemical change on tenderness of cooked meat. Earlier studies indicated that rapid onset of rigor mortis was accompanied by increased rate of glycolysis and net disappearance of adenosine triphosphate, and associated with accentuation of toughness in young birds. Studies were conducted in which glycolysis was absent through chemical inhibition of a glycolytic enzyme, phosphoglyceraldehyde dehydrogenase, and by exhausting the muscle glycogen supply by ante mortem injection of adrenalin. Birds treated thus did not become tough, indicating that a rapid rate of post mortem glycolysis is intimately involved with toughness. Studies are continuing on factors that lead to different glycogen contents in slaughtered chickens and rates of post mortem glycogen disappearance. The extractability of muscle contractile proteins is being studied. Muscle from adrenalin-treated chicken differs from non-treated muscle in solubility characteristics immediately post mortem. While no change occurs during 24-hour aging of the treated muscle, the non-treated muscle changes and becomes quite similar in solubility characteristics to the other.

4. Physiology of Feather Release. The lack of fundamental understanding of the nature of feather retention and release limits major improvements in commercial feather removal. Contract research at Michigan State University has been conducted to conclusion of one phase of study and a new contract has been initiated to follow some

pathways indicated by the completed investigation. Gross anatomical and histological observations were made of the feather, its follicle, and associated muscle and connective tissues. Preliminary investigation of the role of the autonomic and central nervous systems in feather release was conducted, using selective physiologically active compounds. The effect of a slaughter method associated with feather loosening (brain sticking) was studied. Histological studies established the absence of previously postulated circular muscles around the feather follicles, and identified, in the connective tissue surrounding the follicle, elastic fibers which may play a role in feather retention. Data from slaughter tests and the effects of anesthetics pointed to a section of the brain (the medulla oblongata) as the critical area concerned with feather loosening. Selective action on feather release of a series of drugs implicated a functionally separate sub-system of the autonomic or involuntary nervous system. Atropine, which blocks the parasympathetic system, produced marked feather loosening, while tolazoline, which blocks the sympathetic system, had no effect. Other tranquilizers and anesthetic drugs were catalogued as to effects on feather loosening. With this good start, the new contract will be pursued to develop information on histological and biophysical differences between the tightened and relaxed states of feather follicles, the nature of the variation of constrictive force within individual follicles due to chemical and physical agents that may affect feather retention or release, and location of nerve centers that may control feather release.

B. New and Improved Food Products

1. Radiation Preservation of Poultry Products. For its potential value in feeding troops where refrigeration is not available, irradiation-sterilized poultry is considered potentially attractive. With funds transferred from the Department of Defense, studies were conducted to evaluate the usefulness of ionizing radiations in the preservation of poultry products. Irradiation-sterilization of meat and storage at non-refrigerated temperatures causes changes in its flavor, texture, and color. Experiments were conducted to extend knowledge of the problems in irradiation-sterilized chicken and to seek means of preventing or reducing adverse changes. An irradiation dose of 4.5 Mrads has been suggested for sterilization, and the odor and flavor induced by irradiation of chicken parts at room temperature were readily detected at a dose of 0.1 Mrad and increased with increasing dosage. Enzyme inactivation is essential in chicken and other meat to avoid very strong disagreeable flavor changes in subsequent storage. Irradiation of enzyme-inactivated chicken at subzero temperatures reduces the development of off-flavors. Packaging the chicken with a packet of adsorbing charcoal, cooking it in deep fat, and adding seasoning, further mitigate the off-flavor to the degree that negligible adverse reactions would be expected.

Irradiation-sterilized chicken develops an objectionable red color during storage at elevated temperatures in an inert atmosphere if it is not heated prior to treatment. Storage of irradiated chicken at elevated temperatures (room temperature and above) results in exudation of liquid and in a soft, disintegrated texture and dryness in the cooked product. This adverse condition is not controlled by heat treatment for enzyme inactivation and is the most serious remaining problem. Storage at temperatures between freezing and room temperature may help prevent this condition. Support of this research project by the Defense Department will continue at about the same level.

2. Precooked Frozen Foods. Research is conducted on development of the principles governing the behavior of essential basic ingredients that influence their suitability for use in prepared and precooked frozen foods. The peeling tendency of coatings of fried chicken is accentuated in the frozen product. Means of preventing or reducing this defect have been sought. The sequence of processing steps during preparation of fried chicken was found to be of primary importance to adhesion of the coating. Elimination of most of the moisture ordinarily lost during cooking prior to application of the batter produces a product with less tendency to peel than one in which the coating coagulates before the chicken undergoes the shrinking caused by cooking. Tests of the frozen storage stability of foods requiring maintenance of a gel structure were conducted. Simple gelatin gels are unstable below 20° F. Turkey meat loaves, raw and cooked before freezing, and lemon pie fillings are being developed to determine conditions necessary for a stable gel structure. Effects of formulation in connection with processing factors are being investigated. In addition, studies have been initiated on the frozen storage stability of foods whose distinctive character requires maintenance of a foam structure, such as raw or baked soufflés.

C. New and Improved Processing Technology

1. Low-Temperature Microbiology. Fundamental and applied research is conducted to determine growth, survival, and death characteristics of micro-organisms that grow at temperatures of about 32° F. and lower. An extensive review of the literature on low temperature microbiology was conducted, in cooperation with the research program on vegetables at the Western Regional Research Laboratory. The temperature limits of public health hazard and other findings of importance relative to the handling of frozen foods were delineated from published reports and disseminated widely at meetings of industry and public health officers. A temperature gradient incubator was constructed and its use in studies of various factors that affect growth of organisms at chill temperatures was initiated. This device is a practical research tool that determines with great accuracy the

temperature range of growth. Its use has established for a number of cold-tolerant organisms, that temperature of maximum growth is near 95° F. even though they will grow at subfreezing temperatures. No instance has been found in which an organism will grow better below freezing than above. The lowest maxima of this series of tests was a strain of yeast that had a maximum growth near 50° F. Frozen and thawed chicken parts were found to develop spoilage organisms at above zero holding temperatures no faster than parts that had not been frozen. Spoilage was significantly reduced if the parts were dipped for 30 minutes in cold, saturated brine prior to freezing but too much salt was added for good palatability.

2. Tenderness and Other Textural Qualities. The findings of basic research are extended to new and improved processing methods in laboratory investigations. Thus, with the implication of glycolysis in post mortem muscle changes, treatment of poultry flesh with chemicals involved in the glycolytic cycle is a rational approach. Polyphosphates, which occur abundantly in biological systems, play a critical role in glycolysis and were used to treat poultry by a dip in chilled solutions. Effects of this treatment were a change in color of raw product from the normal yellowish cast to a bluish white appearance and a decrease of about 4% in the cooking shrink. The cooked product was normal in appearance and test panels could detect no consistent difference in flavor, tenderness, or juiciness that was related to the treatment. Taste panel tests and objective chemical tests for rancidity made on cooked turkey meat indicated that polyphosphates, incorporated in the prior chilling step, had a stabilizing effect in refrigerated storage (about +40° F.) or in +10° F. frozen storage of the cooked meat. Tests have demonstrated that poultry meat frozen immediately after slaughter and then thawed and aged was less tender than that aged before freezing. The possibility that rate of thawing might be involved in this difference was investigated. Thawing rapidly (4 hours) or slowly (24 hours) had no significant effect on the tenderness of birds aged 3 to 4 days after thawing.

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Area 12 EGGS-- PROCESSING AND PRODUCTS

Problem. The $1.8 billion egg industry is periodically faced with burdensome surpluses that drive prices below the break-even point for many producers. The industry is also faced with a declining per capita consumption. Because eggs used as table eggs have a relatively low elasticity of demand, increased utilization of eggs must come primarily from development of egg-containing products that can compete more successfully on the basis of improved convenience and quality. Adequate knowledge is lacking of the properties, processing characteristics, and new product potentials of eggs needed to develop new markets. Increased utilization of eggs would not only benefit the producer, but would also serve toward eliminating our feed grain surpluses since poultry and egg production account for about one-fourth of all grain fed to animals. Improved egg-containing products would benefit the producer in three ways: by providing an increasingly useful buffer for stabilizing egg prices; by providing additional uses and outlets for eggs; and by providing more remunerative outlets for wholesome eggs that are unsuitable for table use because of appearance or handling characteristics.

Present outlets for the 10% of egg production that is frozen or dried include the baking, confectionery, salad dressing, noodle, and baby food trades. Modified and new products emphasizing quality and convenience are needed to increase acceptance of egg products by these industries and thus to compete successfully with egg substitutes.

At present, principal problems of egg processors exist in four areas. First, improvement in flavor stability, dispersibility, and freedom from pathogenic Salmonella bacteria are needed to realize the potential of yolk-containing solids in convenience foods. Second, improvements in useful properties and lessening of processing costs of egg white products are necessary to alleviate the imbalance and surplus that accumulates as a result of separation of eggs to satisfy the yolk demand. Third, further basic research on egg composition and components is essential to reach an understanding of physical and chemical changes induced by processing and storage and thus provide a rational basis for devising improved processes and products. Fourth, for direct implementation of egg product utilization, formulation studies designed to incorporate eggs into new household and institutional convenience products, are needed. The latter study must encompass a full appraisal of physical, chemical, and microbiological problems peculiar to the formulated products.

USDA PROGRAM

In the Western Utilization Research and Development Division, a broad program of basic and applied research is conducted at the Division headquarters at Albany, California; by contract in Austin, Minnesota; and by grant funds under P.L. 480 in France. Fundamental research is conducted on egg proteins and their relations to the functional properties and quality of eggs, on egg lipids and their role in off-flavor development in yolk solids, on the mechanism of bacterial penetration and survival in eggs, and on the bactericidal, antiseptic, anti-inflammatory, and food preserving properties of lysozymes and other components from eggs. Applied research is conducted on the stabilization of yolk-containing solids to increase the usefulness of eggs in dry mixes and other convenience foods, on new and improved drying procedures to make dried egg fractions and products more readily and more completely dispersible, on various methods of controlling Salmonella in eggs, and on factors in the handling of shell eggs that affect egg product quality and cost.

The Federal program of research in this area totals 13.3 professional man-years. Of this number, 6.9 are assigned to chemical composition and physical properties, 4.0 to new and improved food products, and 2.4 to new and improved processing technology.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations are conducting research on the chemical composition and physical properties of eggs at the rate of 5.5 professional man-years. The programs include a variety of studies on the basic composition of eggs, the nature of various egg proteins, chemical changes involved in thinning of egg white and other deteriorative processes, relation of various egg properties to functional characteristics, effects of bacteria on egg composition, and similar and related topics. About 0.8 professional man-year is devoted to new and improved food products on subjects such as utilization of undergrade shell eggs, new packaging techniques, factors affecting performance properties of eggs, and on antioxidants for food products containing egg yolk. About 0.6 professional man-year is applied to research on methods for preserving eggs, including studies on frozen egg products and enzymatic effects in yolk stabilization.

Industry conducts practically no research on the chemical composition and physical properties of eggs except as incidental to the development of new and improved products and processing technology. It is estimated that egg product manufacturers devote not more than the equivalent of 6 professional man-years to research on control of Salmonella in eggs, on improving performance of dried egg white, and on improving the efficiency of dried egg white manufacture. About 3 professional man-years are devoted to formulation and testing of new

convenience foods containing eggs. Research on improvement of washing, breaking and separating eggs, on improved heat pasteurization equipment, and development of chemical sterilization techniques is conducted at the rate of about 2 professional man-years. About 1 professional man-year is devoted to modification of feeds for laying hens to obtain uniformly deep yolk color in eggs.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Proteins. Light scattering studies have shown that ovalbumin in solution of pH 9 exposed to 95° F. for several days will fail to agglomerate normally when the solution is acidified. Additionally, the coagulation temperature of the ovalbumin in acid solutions is greatly increased. These results are in accord with a mechanism earlier indicated for inferior egg white performance in angel food cake following holding of shell eggs at elevated temperatures.

Work on the possibility that bound cysteine in the yolk is released and translocated into the white, thereby contributing to egg white thinning, was continued. A more efficient recovery method involving dialysis and bromine oxidation for eventual recovery of cysteine-cystine as cysteic acid was developed and applied. Present results, although inconsistent, indicate no measurable cysteine-cystine in the whites from fresh shell eggs. However, when shell eggs are held for several days at 75° F., the bound cysteine decreases in the yolk and increases in the white. No quantitative correlation has, however, been attempted between the translocation of cysteine and egg white thinning.

The use of cellulose column chromatography as a preparative procedure was developed. Several purified egg white proteins were prepared directly from bulk quantities of white, including lysozyme, 3 conalbumins, ovomucoid, 3 ovalbumins, 3 flavoproteins, and a pink protein, possibly a nucleoprotein. The procedure greatly shortens the time required for obtaining isolates and is sharply definitive, and is applicable to the preparation of fairly large quantities (10 grams) of specific components.

Under a research grant to the University of Paris, France, supported by P.L. 480 funds, a project was initiated on lysozymes from various sources, including egg. The objective is to elucidate the relationships between chemical structure and biological activity of lysozymes from various sources as a basis for understanding and utilizing the bactericidal, antiseptic, anti-inflammatory, and food preserving properties of these enzymes. Progress includes a new and effective way of separating and purifying lysozymes, determination of the

sequence of amino acids in egg white lysozyme, and development of a method for studying the enzymatic activity and the specificity of lysozymes using a soluble substrate of these enzymes from Micrococcus lysodeikticus.

2. Lipoproteins. Emulsifying and foaming properties of egg yolk are known to be largely dependent upon the characteristics of the yolk proteins. Previous investigations of yolk proteins, employing combinations of salt fractionation and ether extractions, failed to yield the homogeneous fractions of yolk lipoproteins needed for correlation with functional characteristics of egg solids. Research was undertaken on ultracentrifugation for separation of yolk proteins. Fractions obtained by simple ultracentrifugation of yolk were further separated by ultracentrifugation of various salt solutions of the fractions. In general, the technique did not yield satisfactorily homogeneous fractions and does not appear to be applicable to high-lipid substances such as egg yolk.

3. Fatty Acid Composition of Yolk Lipids. The major fatty acids of egg yolk lipids of hens raised on a typical commercial dietary regime were found to consist of oleic, palmitic, stearic and linoleic acids. The relative amounts of these acids were comparable to previously reported data with palmitic and oleic making up over 75% of the total. Palmitoleic (C_{16}, 1=), arachidonic (C_{20}, 4=), docosohexaenoic (C_{22}, 6=) and docosopentaenoic (C_{22}, 5=) were also detected in minor amounts, and at least twelve other fatty acids, some having branched or odd chains, were detected in trace amounts. Among these, the following were identified: C_{20}, 2=; C_{20}, 3=; C_{20}, 5=; and isomers of the docosohexaenoic and decosopentaenoic fatty acids reported above. Arachidonic, docosohexaenoic and docosopentaenoic acids were isolated in highly purified form.

The total lipids consisted of 69.5% triglyceride, 21% phospholipid and 9.0% minor constituents. At least eighteen components were detected in the minor constituents with carbon numbers from 12.8 to 28.6 as determined by gas-liquid chromatography. However, except for palmitoleic, palmitic, oleic and stearic acids, these occurred in only trace amounts. The triglyceride fraction (69.5% of total lipid) contained 12 different fatty acids with palmitic and oleic making up 83.4% of the total and stearic and linoleic accounting for an additional 13%. The distribution of saturated (S) and unsaturated (U) fatty acids in the triglycerides (G) as determined by a newly developed method was as follows: 1.7% GS_3, 14.1% GU_1S_2, 34.7% GU_2S, and 49.5% GU_3.

About 85% of the total phospholipid consisted of cephalin. However, at least three other components were detected in this fraction. The polyunsaturated fatty acids containing more than two double bonds

were nearly all in the phospholipid and the C_{20} and C_{22} polyunsaturated acids represented about 5% of the total. The ratio of unsaturated to saturated fatty acids in cephalin was close to 1:1, but small amounts of both diunsaturated and disaturated components were also detected.

As the result of this research, the polyunsaturated fatty acids were identified, and their distribution in egg lipid fractions was determined. Since the oxidative decomposition of these acids, in their particular fractions, is undoubtedly a principal factor in off-flavor development, their identification provides a basis for study of the oxidative mechanisms involved.

4. Oxidative Changes in Yolk Lipids. Research was initiated on oxidative changes in yolk lipids as related to off-flavor development. Two volatile decomposition products were found to develop during air storage of yolk-containing egg solids. Further analysis showed that the two products arose from the phospholipid fraction. Surprisingly, infrared spectra of the products showed practically no evidence of carbonyl groups. Analysis of volatiles from sugared yolk-containing egg solids which had undergone oxidative flavor deterioration revealed large amounts of a foreign component which had previously been detected in lesser amounts in the volatiles from unsugared, egg-containing powders stored in air. The component has not yet been identified.

A special type of chromatography applied in the separation of lecithin and cephalin disclosed a new component in the phospholipids of air-stored egg powder. The component was extremely unstable and decomposed rapidly in vacuo. It is postulated that the new component is a precursor of off-flavors and odors.

Cephalin isolated from egg yolk was found to autoxidize much more rapidly than lecithin or highly unsaturated model systems such as methyl docosohexaenoate. Model system studies also showed that egg lecithin has a pronounced effect on reducing oxygen uptake by methyl linoleate, but that cephalin had a pro-oxygenic effect, even though the cephalin was fully hydrogenated to reduce its own contribution to oxygen uptake.

5. Bacterial Spoilage of Shell Eggs. Previous studies have shown that most egg spoilage bacteria are unable to multiply in unsupplemented egg white at its usual storage pH of 9.2. Studies were made, therefore, to determine the mechanism by which eggs spoil bacteriologically. Eggs were experimentally inoculated with various egg spoilage bacteria and then stored at 55° F. Population counts were made on the shell membrane system, on the white, and on the yolk at periodic intervals. Results indicate that the spoilage bacteria

multiply extensively in the shell membrane system before any invasion of the egg contents occurs. Subsequently, there is an increase in bacterial numbers in the white followed by growth in the yolk. This suggests that bacterial spoilage of eggs in storage may be controlled if storage conditions are selected to minimize or eliminate growth of bacteria in the shell membrane system.

Earlier laboratory studies showed that the presence of soluble iron in wash water greatly influences the rate and extent of spoilage of shell eggs by Pseudomonas. Similar results were found with other common gram-negative egg spoilage bacteria including Proteus, Alcaligenes, Aerobacter, Paracolobactrum, Achromobacter, and Salmonella. The effect of iron in wash water for shell eggs in increasing Pseudomonas spoilage has been demonstrated under actual ranch conditions. Thus, on one ranch with no previous history of "green rot" trouble, raising the iron content of the wash water from 0.4 to 10 ppm increased the rots detected after 48 days' storage at 55° F. from 0.8 to 2.5%. On a second farm where a serious spoilage problem existed, replacing the high iron wash water (4.8 ppm) with one of much lower iron content (0.2 ppm) decreased the incidence of rots from 6.2 to 0.8% after a similar storage period.

B. New and Improved Products

1. Mixtures of Carbohydrates and Yolk-Containing Solids. Studies on the stabilization of yolk-containing solids against oxidative flavor deterioration were completed. It was shown that carbohydrates, i.e., sucrose and corn syrup solids varying in degree of dextrose equivalent, provided good stability when added to yolk-containing egg solids. In fact, results showed that for every type of yolk-containing solid of commercial import, it is possible to achieve a good combination of flavor stability, performance value and stability, and sweetness by selection of type and level of added carbohydrate. Amount of carbohydrate to be used is critical. With increasing levels of carbohydrate, a point of maximum flavor stability was reached, but at slightly higher levels, abrupt and marked flavor instability was found. For various carbohydrates, the levels at which maximum flavor stability and instability were observed were found to be related to the average molecular size of the particular carbohydrate. Additionally, the levels of added carbohydrate yielding optimal flavor stability were correlated with the state of emulsion in the powder. Retention of the fat in a finely dispersed state led to marked flavor instability.

In the course of the research on yolk-containing solids, it was found that one reason for the variability of spray-dried yolk lies in the extent of protein alteration induced by processing. The protein alteration may be induced at three stages: (1) during preheating of the liquid prior to drying, (2) during high-heat spray drying, and

(3) during storage of the product at high temperatures. Analytical methods were developed for appraising the extent of protein alteration.

C. New and Improved Processing Technology

1. Control of Salmonella in Egg Whites. After preliminary screening of 28 bacterial strains for their usefulness in deglucosing egg white prior to drying, six were selected as the most promising and were examined over a wider range of nutritional conditions. Those selected were A. aerogenes B 199, E. coli. H 23, S. lactis E 71, S. cremoris B 634, L. mesenteroides B 641, and Ped. cerevisiae B 1345. E coli. H 23 is an organism having additional value in being inhibitory to Salmonella. Of these organisms, only L. mesenteroides B 641 deglucosed egg white satisfactorily at pH 5.0 but required nutritional supplementation. It was then considered that fermentation at low pH might be of value in controlling Salmonella development. In further studies, it was found that three additional organisms, M. freundenreichii B 2354, S. lactis B 633, and L. casei B 442 would also deglucose whites satisfactorily at pH 5.0 providing acidification was made with citric acid and the egg white was supplemented with low levels of yeast extract and casein hydrolysate.

Tests made with one strain, Salmonella typhimurium TMI, have shown that in pure culture in egg white, the organism grows out well over a pH range of 5.0 to 7.0. In mixed culture, it grew out competitively at pH 7.0 with several of the above organisms, but was unable to compete at pH 5.0 with L. mesenteroides, M. freundenreichii, or S. lactis. The latter organisms multiplied normally and in doing so appeared to exert a bacteriostatic effect on the S. typhimurium. Low pH fermentation of egg white appears to have potential value in controlling Salmonella.

2. Effect of Age of Layer on Egg Quality. It has been previously reported that eggs from older layers, though exhibiting defects as table eggs, are advantageous for conversion to egg products because they yield a substantially higher percentage of their total content as yolk. Price considerations are such that the total value of the contents of eggs from older layers would be 10 to 15% greater than that from younger layers. These findings have been confirmed with eggs from two different White Leghorn strains. Additionally, it has been found that performance value of yolks from older layers as evaluated in sponge and layer cakes is equivalent or slightly superior to that from younger layers. The egg whites from older layers, however, have been shown to be low in solids and to yield angel food cakes with volumes consistently lower by 3 to 4% than those obtained with whites from younger layers. The difference decreases with holding of the shell eggs and is not apparent two to three weeks after lay.

The relative value for table and breaking stock of eggs from layers uniformly forced into their second year of lay by a controlled "force molting" process was appraised with a flock of approximately 15,000 layers. The birds were all of the same hatch date and were forced into molt at 21.5 months of age. The study was carried on for 8 months after molting. Results suggest that egg quality as measured by internal albumin quality, uniformity thereof, and shell strength is satisfactory for table grade eggs only for about the first three months of the second year of lay. After this time, the eggs resemble those laid at the end of the first year of lay and, similarly, offer particular advantages for egg breaking stock such as size, yolk yield, and yolk solids content.

3. Improved Dispersibility of Dry Egg Products. Further progress was made on the development of a modified spray-drying procedure for yielding instantly dispersible yolk-containing solids. A carbonating device was designed, constructed, and tested successfully in a 24-foot commercial pilot dryer. The product possessed excellent initial quality as well as dispersibility and is presently being compared with instant products made by agglomeration and by hot air drying of mechanically preformed foams. These studies are continuing.

Instantizing methods have also been applied toward improvement of the dispersibility of dried egg whites. Drying techniques other than conventional spray drying show promise. Spray drying of larger particles, gas impregnation of the white prior to spray drying, forced air drying of mechanically preformed foams, and agglomeration are all helpful, but in some cases only where carbohydrates have been added or when the whites have been concentrated prior to drying. In general, egg white, despite the absence of lipids, is more difficult to instantize than yolk-containing products.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

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New and Improved Products

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New and Improved Processing Technology

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1/ Research conducted under P.L. 480

Area 13 PHARMACOLOGY

Problem. Advances in agricultural science and processing technology necessitate the use of an increasing number of new chemical compounds whose safety must be established. The mutual interests of both the producers and the consumers of agricultural products, demand that public agencies participate in securing unequivocal evidence of safety before products of advanced technology are marketed. This responsibility is particularly acute where a public agency, such as the Department of Agriculture, contributes to technological developments that result in intentional or unintentional addition of untested components in foods, feeds, or in materials contacting the persons of consumers, or developments that result in the introduction, concentration, or modification of natural components in a way that may have an adverse physiological effect on consumers. Types of materials that require continuing surveillance include food additives, inadvertent residues of pesticides and other useful agricultural chemicals, antibiotics and medicinals, and the naturally occurring chemical constituents of physiological importance. In the areas of interest to Agricultural Utilization Research and, in particular, in connection with process and product developments of the four Utilization Research Divisions, such compounds must be tested by short- and long-term ingestion in experimental animals, such as rats and dogs, to secure toxicological data required by the Federal Food and Drug Administration to establish safety and legal certification for their use. The unequivocal establishment of safety for any useful chemical involves much more than merely conducting animal feeding tests on a routine service basis. It often requires the development of original chemical analytical procedures and metabolic fate studies in experimental animals, as well as new methodology and observational techniques for studies of new chemicals. Each assignment in this field is a new area for original, often very fundamental research.

USDA PROGRAM

Pharmacological investigations supporting the Department's utilization research and development program are conducted in the Western Utilization Research and Development Division at Albany, California. Agricultural products and additives required to preserve or otherwise treat them are investigated as they may cause toxic or allergenic reactions. Laboratory methods for discovering the metabolic fate of chemical compounds in animal physiology are developed and applied to problems in the utilization of farm products. Plant constituents that exert deleterious or beneficial effects on animal growth are studied to determine quantitative responses.

The Federal program of research in this area totals 9.0 professional man-years assigned to pharmacology investigations. Additional pharmacological studies directed toward specific commodities are reported elsewhere and include 4.0 professional man-years assigned to chemical composition, physical and physiological properties of castor, safflower, and other western oilseeds (reported in Area No. 9 of A Summary of Current Program, 7/1/62; and Preliminary Report of Progress for 7/1/60 to 6/30/62); and 0.5 professional man-years support for an employee, whose salary is provided by the Dried Fruit Research Advisory Committee, assigned to chemical composition and physical properties of deciduous fruit and tree nuts (reported in Area No. 6).

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported no program within this area. Related programs were reported specifically in connection with the agricultural commodities concerned.

Industry and other organizations including consulting laboratories and pharmaceutical and chemical companies conduct pharmacological tests which are required before new drugs and chemicals can be sold as food or feed additives. Smaller organizations utilize the services provided by some six major toxicological laboratories in the U.S. for tests on the safety of compounds they develop. There are undoubtedly other such facilities which are smaller in size but capable of performing similar studies. Long-term tests usually cost in the neighborhood of $100,000 per compound. The research effort that may be directly related to the improved utilization of farm products is estimated as being equivalent to about 60 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Pharmacology Investigations

1. Gossypol in Cottonseed. Gossypol, a constituent of cottonseed, restricts the use of cottonseed meal for feed purposes because of its toxicity. Study of the relationships of gossypol with animal metabolism, particularly with lysine availability and detoxification mechanisms are in progress. The metabolic fate of gossypol has not yet been established because typical phenolic breakdown products cannot be identified in excreta by methods available at this time. Detoxification of gossypol by rumen fermentation in ruminant animals is considered a possibility and evidence was obtained that the compound can be degraded by microbial fermentation in laboratory experiments. Studies on chemical alterations of gossypol to reduce its toxicity indicate that some antioxidants can enhance the toxicity of gossypol. On the other hand, oxidizing compounds added to the animal diet, decrease the toxicity.

2. Estrogens from Plant Sources. Estrogens (compounds exhibiting female hormone activity) have been discovered in plant materials including vegetable oils. Corn, soybean, rice, wheat germ, peanut, and sesame oils were found to exhibit estrogen-like properties. Attempts to isolate and identify active constituents have not yet been successful. Cottonseed oil was very weakly active. Legumes are found to be a source of estrogens and samples of dehydrated alfalfa have been tested in connection with forage research investigations to locate active compounds such as coumestrol and indicate quantitatively the hormonal activity. Despite published accounts of a high degree of estrogenic activity in hop oils, no such activity was encountered in five different samples investigated.

3. Physiological Activity of Dry Beans. Studies of the effects of dry bean constituents on the action and condition of intestines of laboratory animals are conducted in relation to the flatulence problem of dry bean ingestion. Hyperemia (reddening) of the small intestine of rats and mice has been observed following ingestion of uncooked beans. Injection of certain uncooked bean components into the intestines has had a similar result, sometimes accompanied by increased peristalsis, heavy mucous production, distension, and turgidity. Saponins isolated from beans have been somewhat active in this regard. Extracts of beans obtained with Skelly F (a petroleum fraction), benzene, and ethyl acetate do not cause the intestinal irritation when injected into tied-off loops of rat intestine. However, the extract of beans with 60% ethyl alcohol gives a very active product. This extract contains, among other compounds, S-methyl-L-cysteine which is an amino acid commonly found in beans. A purified sample of this compound also proved irritating when injected into loops of rat intestine. Similar irritations were observed when extracts from cooked beans were used.

4. Trypsin Inhibitor of Raw Soybeans. Raw soybeans contain a trypsin inhibitor that can be inactivated by heat treatment. The action of raw soybeans in animal diets is to inhibit growth and cause enlargement of the pancreas. Studies were conducted to bring light upon the specific activity of the inhibition. At a 4% level in the diet, no effect on pancreas size was noted; but a 60% increase in pancreas weight was observed at the 8 and 16% level in diets. Protein levels were maintained constant by additional crude casein to the 14% level. Preliminary tests have been conducted to determine whether isolated soybean inhibitor will exert the same effects as raw soybean meal (growth inhibition, decreased food efficiency, and pancreas enlargement) or whether there are other factors in soybeans that are involved. A 35-day feeding test at two levels of commercially prepared trypsin inhibitor produced all three effects. Mold-fermented soybean product (tempeh) was evaluated in short-term feeding tests. Trypsin inhibitor is inactivated by heat in the process and no pancreatic enlargement

was observed. Rat growth was inferior on tempeh and protein efficiency was reduced compared with casein as a protein source.

5. Peanut Hemostatic Factor. Evidence has been presented showing that peanuts exert a hemostatic effect and offer relief for hemophiliacs. As yet a suitable assay method is needed to aid in isolation of the factor or factors involved. A pronounced constrictor action on smooth muscle has been demonstrated in an absolute-alcohol extract of defatted, skinless peanuts. This effect may be related to the hemostasis and can be measured by the reaction of excised guinea pig colon tissue. Chemical isolation of constituents of the alcohol extract revealed the presence of p-coumaric, p-hydroxy-benzoic, and probably phloretic acids.

6. Feed Value Studies of Replacement Crops. The commercial value of crops introduced to replace those now in surplus may depend upon livestock feed values of residues such as defatted seed meal. Enough toxicity or growth inhibition effects have been demonstrated to emphasize the continuing need for pharmacology testing of all such replacement crops. Sesbania macrocarpa (a potential plant protein source) contains a high level of the amino acid canavanine, reported to exert antimetabolic effects in chicks. Very poor growth resulted when chicks were offered feed with 35% Sesbania seed. Palatability factors may be involved, and no toxic effects were observed in preliminary short-term feeding tests with mice. Crotolaria intermedia (a source of industrial type gum and a potential plant protein source) was highly toxic to rats. Fed at the 5% level it caused death in 35 days; at 1%, no deaths were caused but growth was inhibited. Vernonia anthelmintica (an oilseed) has been used as a vermifuge. When it was fed at the 20% level, it inhibited rat growth. Defatted meal of Lunaria annua (an oilseed) showed promise as a livestock feed after treatment to remove isothiocyanate. Defatted jojoba seed (a source of a potentially useful liquid wax) fed at the 3% level was toxic to rats, resulting in poor growth, reduced food intake, and testicular atrophy. A palatability factor may be involved as no harmful effects were noted if the material was given by stomach tube. Mustard seed meal and oil were both tested for effects when fed to rats. The meal at 20% and oil at 0.1% permitted normal growth. Higher levels of the oil inhibited growth. Utilization research to obtain useful byproduct feeds from these crops is clearly indicated.

7. Detoxification Mechanisms. Natural chemical mechanisms in animal metabolism exist to detoxify potentially harmful substances. By study of the metabolic fate of ingested chemicals, an important new mechanism was discovered. Phenolic compounds can be detoxified by methylation (attachment of a methyl group to the hydroxyl radical on the benzene ring). This mechanism depends upon a suitable source of methyl groups for the methylation, which is found in choline and methionine. Two important aspects of this finding are (1) the importance of methyl

sources in basal diets where compounds being tested may depend upon this detoxification method, and (2) the possibility of creating choline deficiency where desirable for biochemical studies by use of added phenolic substances that will use the existing choline by this detoxification mechanism. In addition, a unique method of metabolizing the phloroglucinol ring of flavonoids by splitting the ring was observed. Such a ring fission is rare in mammals, although it is commonly accomplished by microorganisms. Procedures for making such metabolic fate studies are being advanced and applied to an increased number of compounds of agricultural interest. The following of the metabolic fate of ingested compounds as a means for establishing their safety or toxicity is a positive approach in contrast to standard toxicity testing, which is empirical, depending upon clinical and post mortem evaluation of the effects of ingesting the compound in question when fed at different levels. After feeding, urine is collected and analyzed for products of metabolism. For example, a major metabolite, not found in normal rat urine was found after the animals were fed a diet containing 2% of the spice, sage. From this result it can be predicted that a precursor compound existed in the sage. Fractionation could be used to identify the compound if desirable. Then the possible effects of such a compound on growth rate and other factors could be determined using a purified isolate of the precursor in feeding tests.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Pharmacology Investigations

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Area 14 CROSS COMMODITY--BASIC STUDIES (by State Experiment Stations)

Problem. Basic problems confronting the food industry center around providing the scientific data which will be needed to meet the challenges of converting our food supply into high-quality products of improved appeal to consumers at the lowest possible cost. Solution of these problems will depend upon application of the fundamentals of chemistry, physics, engineering, and microbiology to the production, preparation, processing, packaging, storage, distribution, and utilization of foods.

Basic research on chemical composition, physical properties, enzyme activity, pigments and color is needed to lay the groundwork for new product development and improved processing techniques. An increasing number of new products is being introduced and many of these are urgently in need of improvement which will depend upon basic work for guidance. Trends in individual foods such as portion control, changes in form and changes in market goals will need to be founded on sound principles if they are to be successful. For example, we now have a complete line of foods for the very young and, in the future, it is quite likely that we will have a complete line of foods for the very old.

Product innovations carry with them the need to improve actual processing procedures. Basic problems involved in heat transfer, fluid flow and the use of forms of energy other than heat in processing await solution. Control of microbiological activity is one of the major objectives of processing. Rate of bacterial growth, thermal inactivation of bacterial spores and method of utilizing the desirable properties of micro-organisms all require additional study. Research is needed to point the way to improved high-temperature short-time sterilizing processes, sterilization or pasteurization by irradiation, dehydrofreezing, freeze drying and freezing. The need for continuous processing, automation and the demand for a year-round supply of standardized uniformly high-quality products will necessitate invention of new processing machinery and techniques.

Foods must be made not only more nutritious but more appealing to the consumer. Problems in the area of developing or creating food flavors must be solved before full advantage may be taken of changing trends in consumer tastes. Evaluation and modification of the basic genetic make-up of food plants and animals from the standpoint of taste, quality and nutritive value is a continuous process. Information is also needed on the characteristics and properties of by-products and residues which accumulate at processing plants in order

to develop new products, extend markets, create new outlets or provide salable products of economic benefit.

PROGRAM OF STATE EXPERIMENT STATIONS

In 1961 the State Experiment Stations reported as follows:

Chemical Composition and Physical Properties
(18.7 professional man-years)

The stations have a large program of research which deals with basic chemical composition and physical properties of different foods. Fundamental chemical composition and physical property data are collected because of their relation to and use in understanding changes which occur in market channels, effects of thermal processing or quality loss. Basic work on enzymes, enzyme systems, substrates and reaction products, kinetics, isolation and characterization is pursued extensively because the information is needed to control discoloration and other enzymatic changes and to facilitate establishing processing conditions. Polyphenolic constituents are under intensive study. Color and pigment studies embrace flavonoid pigments and their degradation in processing, measurement of retention of anthocyanin pigments in processed fruits and vegetables, biosynthesis and stability of carotenoids, and measurement of color. The biochemistry of physiological effects of plant constituents; redox-potential in fermentation and aging of food products; and mechanism of lipid oxidation are specific problems under investigation. Other work deals with methods of analysis, chemistry of pectin and pectic enzymes, deteriorative changes and off-flavor development including role of lipids in food flavors.

New and Improved Food Products
(12.4 professional man-years)

Various aspects of the underlying principles of new product development are under investigation. Economic means for utilizing crops, formulation of new products, introducing variety or convenience and improving the quality of existing foods are logical goals. Techniques are not always equally successful. Product requirements and consumer wants vary. New items such as instant applesauce, precooked, convenient poultry and egg foods and fresh and frozen shrimp products are under development. Specialty items suitable for canning and which can be made from local products are of interest. Bakery mixes are popular and researchers seek to develop a complete, highly acceptable corn meal mix. Basic concepts and procedures governing product formulation are being explored and a program which is designed to test the adequacy and effectiveness of procedures useful in securing adoption of station new product developments is in progress. Another program involves technical assistance to food processors which aids them in commercial introduction of food products.

New and Improved Food Processing Technology
(15.2 professional man-years)
Basic programs designed to accumulate data on new and improved processing technology are conducted with several different objectives in mind. Heat transfer and flow characteristics in liquid food products arebeing actively researched. Another program involves study of the steps in juice extraction and manufacture including use of electronic devices and controls in food plant operations. Preservation of foods by means of ionizing radiations and the effect of irradiation on nutritive value, quality, color, and texture of foods is under intensive investigation. Use of sub-sterilizing doses is a promising avenue of work and radiation pasteurization of seafoods is receiving attention. Newer techniques of dehydration, such as freeze-drying, are being tested and basic data accumulated. The effect of various means of processing on the microscopic structure of foods is being evaluated. Basic studies designed to improve processing and process techniques are underway for: curing and smoking turkeys; freezing of meats, fruits and vegetables; characterization of effects of low temperatures on cellular properties of food products; chemical preservation, disinfection and sterilization of foods and food containers; and blanching, packaging and storage. These principles must be established before new techniques may get widespread use. Development of specialized food processing machinery designed to handle specific jobs in processing food products is also in progress.

Flavor of Foods
(6.0 professional man-years)
Flavor analysis is receiving increasing attention through efforts to identify flavor components, their precursors and heat induced flavors. Development of new and improved methods for the collection, separation, identification and analysis of the flavor components has opened the door to many new investigations designed to determine the significance of various compounds in the flavor of foods. Integration of these findings with similar sensory and consumer tests is an important phase of this evaluation. Product formulation, treatments and additives must be evaluated for effects on flavor of finished products. Data are accumulating on precursors, on changes due to handling, storage and processing, and on the effects of production practices on flavor.

Microbiology of Foods
(13.7 professional man-years)
Fundamental research on the microbiology of foods encompasses work on: microbiological fermentations and enzyme systems; thermal-death kinetics and their relationship to thermoprocessing; microbial spoilage; ecology, taxonomy, physiology and nutrition of micro-organisms of importance to the food industry; sanitation and control; techniques for identification of bacteria; and, use of micro-organisms as

analytical tools. Different aspects of problems associated with bacterial endospores, their thermal resistance and the relationship of this resistance to thermoprocessing and spoilage of thermally processed foods are being investigated. Another phase of these studies involve attempts to use heat shocking as a means for thermal activation of spores and their subsequent destruction by moist or dry heat. The effect of heat in the presence of food components and the nutrient media employed are receiving special attention. Another approach involves designing and using very specialized apparatus to study the thermal resistance of both vegetative cells and spores of micro-organisms in the presence of both moist and dry heat. The proper processing times, temperatures and procedures are being re-evaluated using Bacillus thermoacidurans as the test organism. Many cleaning, sanitation and food spoilage problems are being tied to specific microbiological causes. Synthetic activity, physiology and effect of low temperature of organisms under both aerobic and anearobic conditions are studied. Other work includes use of micro-organisms as special analytical tools. Methods of isolation, examination, identification and quantitation of microbial populations found in frozen foods are being sought. Microbial contamination during processing is determined and work on identification of bacteria by gas chromatography is in progress. Portions of this fundamental program receive support from foundations and industry. Some projects receive USDA support.

Varietal Processing Adaptability
(1.5 professional man-years)

Developing the specific requirements and testing the suitability of plant varieties and breeding selections for processing is an important and continuing program. Specifications for processing quality vary with crop and process to be used. It is necessary to develop guides and provide recommendations for processing crops grown within state boundaries. Portions of this work are done in cooperation with USDA.

New and Improved Industrial Products and Feeds from By-products (including Waste Disposal)
(1.0 professional man-years)

Research is in progress which is designed to provide underlying principles and basic data useful to by-product utilization. This work involves development of recovery techniques, evaluation of nutritional qualities, storage stability, material balance, and physical form studies which determine whether products will be acceptable and economically feasible.

Area 15 REPLACEMENT CROPS-- UTILIZATION POTENTIAL

Problem. Farmers could achieve more economic use of their land if new and profitable crops were available for their choice that would have different end-use patterns from those presently grown. For example, it would be advantageous to develop a new oilseed crop yielding unique fatty acids that could find industrial use in applications for which acids from presently available domestic oilseed crops are unsuitable. To develop a new crop, three basic steps are involved: (1) survey of wild plants, in cooperation with plant scientists, to identify those having both potentially valuable components and promising agronomic potential for use in the U. S.; (2) detailed physical and chemical characterization of components of interest to obtain clues to likely end uses; (3) selection of the most promising species followed by additional utilization research to explore uses and demonstrate industrial potential and by additional agronomic research to establish proper cultural practices and select the best strains and varieties. Only after these steps have been successfully accomplished can a proposed new crop be offered to agriculture and industry for introduction and development. Obviously, a program of this type is a long-range one. Yet, whether the future of agriculture involves conditions of surplus, of greater emphasis on foods and feeds, or of necessity for greater national self-sufficiency, the nation will benefit from availability of optimum, practical crop plants to serve its needs.

To achieve the objective, survey and characterization work needs to be greatly increased, since the greater the number of species examined, the greater will be the opportunities for finding plants meeting the criteria of high utilization and agronomic potential. Work of the Department has already revealed several promising sources of new potentially valuable water-soluble gums, pulp fibers, and oils containing unique fatty acids such as hydroxy-unsaturated acids, capric acid, epoxidized acids, and unusual long-chain fatty acids. In order to demonstrate the potential of these new materials, further work is required on their physical and chemical properties and reactions, on processing to obtain maximum recovery from source plants, and on by-products from processing, such as oilseed meals.

USDA PROGRAM

Basic and applied research is being conducted on hydroxy-unsaturated acid-containing oilseeds, in the Western Utilization Research and Development Division's headquarters laboratory at Albany, California.

The basic, compositional studies emphasize the development of special analytical techniques for application to new oils containing hydroxy-unsaturated fatty acids. In the applied area, research is conducted to develop and evaluate industrial products from the hydroxy-unsaturated oils.

The Federal program of research in this area totals 4.0 professional man-years. Of this total, 2.0 are assigned to chemical composition and physical properties; and 2.0 to industrial utilization.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

Related program of all State Experiment Stations and industry and other organizations is reported by the Northern Utilization Research and Development Division in Summary of Current Program and Preliminary Report of Progress.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Dimorphotheca and Lesquerella Seed Oils. Dimorphotheca oil has as its major component acid (about 65%), 9-hydroxy-10,12-octadecadienoic acid (dimorphecolic acid). Lesquerella oil has as its major component acid (about 60%), 14-hydroxy-11-eicosenoic acid (lesquerolic acid). Improved methods have been developed for processing experimental quantities of these oilseeds to provide sufficient oil for study in the laboratory. Analytical studies have been carried out, extensively on Dimorphotheca, and to a much lesser extent on Lesquerella seed (which was more recently assigned for exploratory study). Thirty-eight samples of Dimorphotheca seed of four different species (annua, aurantiaca, calendulacea and sinuata) grown by Crops Research Division in ten different locations in the United States, were analyzed for their contents of major components, particularly oil and meal in the seed, and hydroxy-conjugated-dienoic acid in the oil. Composition of these samples varied widely, with as little as 3.70 to as much as 14.84% of oil in the seeds, and from about 27 to 65% of conjugated-dienoic acid in the oils. There was no discernible relation of oil content or composition to seed species, and no simple relation between the content of conjugated-dienoic acid in the oil and the oil content of the seed. Methods previously developed for the analysis of the component fatty acids of castor oil are being adapted for the analysis of Dimorphotheca and Lesquerella oils. It appears that silicic acid column chromatography in combination with, and followed by, gas-liquid chromatography will afford satisfactory results. An analysis of Dimorphotheca oil yielded the following composition: 31.7% nonhydroxy, 65.1% monohydroxy, 0.75%

dihydroxy, and 2.5% ketodienoic acids. A semi-quantitative method was developed for the determination of non-conjugated hydroxy fatty acids in fatty materials. This method is based on the characteristic near infrared (2.7 - 2.8 microns) absorption exhibited by hydroxy compounds, and is proving valuable in routine use. A method was developed for the preparation of small amounts of pure dimorphecolic acid, as the methyl ester. The ester was prepared in highly purified form for use as a reference compound and its physical characteristics have been determined. Larger amounts of methyl dimorphecolate in purity suitable for use in studies on chemical modification can conveniently be prepared by partitioning between two mutually immiscible solvents such as commercial pentane and acetonitrile. In this way more than 90% of the methyl dimorphecolate present in the raw mixture of the methyl esters of Dimorphotheca oil can be conveniently recovered in better than 90% purity.

B. Industrial Utilization

1. Industrial Products from Hydroxy-unsaturated Oils. A system was developed for hydrogenating methyl dimorphecolate which provides quantitative conversion to the hydroxystearate in a very pure, crystalline form without recrystallization. This was in marked contrast to hydrogenation systems previously used which gave relatively large proportions of isomerization and hydrogenolysis products. Studies were undertaken to prepare a series of phosphorus-containing esters of methyl dimorphecolate. Initial experiments were conducted by reacting methyl dimorphecolate with phosphorus trichloride using dimethyl aniline as a hydrochloric acid acceptor. This reaction required such severe conditions that excessive dehydration occurred. More recently both methyl dimorphecolate and lesquerolate were satisfactorily phorphorylated by reaction with diethyl phosphorochloridate under suitably controlled conditions. Purification was effected by partitioning the products between acetonitrile and Skellysolve F. The same partitioning system was used to purify long chain phosphite diesters produced by the reaction, at elevated temperature, of methyl lesquerolate and dimethyl phosphite. The analogous reaction with methyl dimorphecolate was not successful owing to its dehydration to the triene. These new procedures are proving to be very useful for the preparation of phosphorus-containing esters of the long chain hydroxy acids, which work is being continued. The utility of Dimorphotheca and Lesquerella oils for preparing urethane-type foams was investigated. When mixtures of these oils with triisopropanol amine were used as the polyol component, strong, lightweight, fluorocarbon-blown foams were obtained. These foams were slightly yellow, but otherwise were very similar to castor oil-based foams. No indication was obtained of an increase in compressive strength which might be expected if the conjugated diene in the dimorphecolate

moiety polymerized on aging (4 months). It would appear entirely feasible to use these oils in urethane-type foam formulations. The utility of Dimorphotheca oil in surface coatings is being evaluated, cooperatively, at North Dakota State University. Preliminary results show considerable promise for this oil in surface coatings. Although slow drying, the oil exhibits final film characteristics desired in such products.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical Composition and Physical Properties

Goldblatt, L. A. 1962. Processing Oilseeds. Proceedings of Pacific Northwest Oilseed Crop Conference. Oregon State University, Corvallis, Oregon. pp. 58-72.

Line Project Check List -- Reporting Period July 1, 1960 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Summary of Progress	Incl. in Area & Sub-Subheading
W1 2-24 (Rev.)	Air classified flours	Albany, Calif.	Yes	1-A-1 1-B-4
W1 2-26[1]	Gliadin and gliadin derivatives	Albany, Calif.	Yes	1-A-11
W1 2-27	Wheat feed products	Albany, Calif.	Yes	1-E-1
W1 2-29	Bread flavors	Albany, Calif.	Yes	1-A-15
W1 2-34 (C)[1]	Upgrade wheat and barley for feed	Pullman, Wash.	Yes	1-E-1
W1 2-36	Lipoglutenin studies	Albany, Calif.	Yes	1-A-4
W1 2-37[1]	Flavor precursors	Albany, Calif.	Yes	1-A-15
W1 2-38	Parboiled wheat foods	Albany, Calif.	Yes	1-B-1
W1 2-39 (C)	Effect of additives on bread flavor	Cambridge, Mass.	Yes	1-A-16
W1 2-40 (C)	Estrogens in wheat germ and bran	Ames, Iowa	Yes	1-D-1
W1 2-41	Wheat endosperm constituents	Albany, Calif.	Yes	1-A-6
W1 2-42 (C)	Methods for determining wheat proteins	Pullman, Wash.	Yes	1-A-1
W1 2-43	Gluten foods	Albany, Calif.	Yes	1-B-2
W1 2-44	Protein interactions	Albany, Calif.	Yes	1-A-9
W1 2-45 (C)	Water-dispersible protein preparations	Lafayette, Indiana	No [2]	
W1 2-46 (C)	Chemical basis for cohesiveness in gluten	Kansas City, Missouri	No [2]	
OCDM-0-1	Fallout shelter foods	Albany, Calif.	Yes	1-B-6
UR-A10-(10)-22	Rheology of wheat flour doughs	Haifa, Israel	Yes	1-A-10
UR-E9-(10)-2	Composition of whole wheat lipids	Paris, France	Yes	1-A-5
UR-E9-(10)-7	Immunochemical analysis of wheat and barley proteins	Paris, France	Yes	1-A-3
UR-E9-(10)-8	Solubility of wheat gluten proteins	Montpellier, France	Yes	1-A-14
UR-E9-(10)-43	Phosphorus in wheat flour	Paris, France	Yes	1-A-13
UR-E9-(10)-44	Ultrasonic study of wheat gluten	Paris, France	Yes	1-A-12
UR-E9-(10)-45	Enzyme action in low-moisture grain	Paris, France	Yes	1-A-8
UR-E15-(10)-31	Wheat germ proteins	Bologna, Italy	Yes	1-A-7
UR-E21-(10)-1	Sulfhydryl groups in wheat	Poznan, Poland	Yes	1-A-2
UR-E29-(10)-14	Wheat flour lipids	Chorleywood, England	Yes	1-B-5
UR-E29-(10)-47	Biological value of processed wheat	Cambridge, England	Yes	1-B-3

[1] Project completed during reporting period.

[2] Recently initiated contract project, no progress reported

Line Project Check List--Reporting Period July 1, 1960 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Summary of Progress	Incl. in Area & Sub-Subheading
Wl 2-31	Effects of processing on properties and new product development	Albany, Calif.	Yes	2-A-1 2-B-1 2-B-2 2-C-4
Wl 2-32[1]	Rice preprocessing behavior	Albany, Calif.	Yes	2-C-2
Wl 2-33[1]	World rices evaluation	Albany, Calif.	Yes	2-C-3
Wl 2-35[1]	Rice drying	Albany, Calif.	Yes	2-C-1
Wl 3-13[1]	Physiologically detrimental components of forages	Albany, Calif.	Yes	3-A-1 3-A-2 3-A-4
Wl 3-14[1]	Forage polysaccharides and organic acids	Albany, Calif.	Yes	3-A-6
Wl 3-16 (Rev.)	Improved forage feed products	Albany, Calif.	Yes	3-A-4 3-B-1
Wl 3-17 (C)	Interaction of forage antioxidants	Berkeley, Calif.	Yes	3-A-5
Wl 3-18	Phenolic components of forages	Albany, Calif.	Yes	3-A-1 3-A-2 3-A-3
UR-E29-(10)-52	Structure of alfalfa polysaccharides	Edinburgh, Scotland	Yes	3-A-6

[1] Project completed during reporting period.

Line Project Check List -- Reporting Period July 1, 1960 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-Subheading
W2 2-7 (Rev.2)	Molecular properties of wool and mohair proteins	Albany, Calif.	Yes	4-A-2
W2 2-10 (Rev.)[1]	Cause and prevention of yellowing in wool	Albany, Calif.	Yes	4-A-1
W2 2-11 (Rev.)	Minimizing degradation of wool by acids and alkali	Albany, Calif.	Yes	4-A-2
W2 2-18[1]	Engineering laboratory studies of processing treatments to reduce shrinkage in wool products	Albany, Calif.	Yes	4-B-1
W2 2-21 (C)[1]	Finishing treatments for wool fabrics treated with epoxy-polyamide resin	Dedham, Mass.	Yes	4-B-1
W2 2-22	Chemical treatment of wool for shrink resistance and other "easy care" properties	Albany, Calif.	Yes	4-B-1 4-C-1
W2 2-23 (C)[1]	Thermal stability of wools and mohair	Salt Lake City, Utah	Yes	4-A-1
W2 2-24	Effect of fabric construction and functional properties	Albany, Calif.	Yes	4-C-3
W2 2-25	Fulling properties of wool fabrics	Albany, Calif.	Yes	4-B-4 4-C-2
W2 2-26	Setting and relaxation of fibers in wool fabrics	Albany, Calif.	Yes	4-B-3
W2 2-27	Uniformity and strength of wool yarns	Albany, Calif.	Yes	4-C-4
W2 2-28	Mechanical behavior of wool fibers and fibrous assemblages	Albany, Calif.	Yes	4-A-3 4-A-5
W2 2-29	Effects of radiation on natural and modified wools	Albany, Calif.	Yes	4-A-1 4-A-4
W2 2-30	Nuclear magnetic resonance absorption of natural and modified wool and mohair	Albany, Calif.	Yes	4-A-2
W2 2-31 (C)	Chemical processes for removing vegetable matter in wool	Washington, D. C.	Yes	4-B-5
W2 2-32	New types of yarns and fabrics from coarse wools	Albany, Calif.	Yes	4-C-4 4-C-5
W2 2-33 (C)	Improved bleaching of wool	Lowell, Mass.	Yes	4-B-6
UR-E8-(20)-10	Finishing treatments for improved qualities in wool fabrics	Helsinki, Finland	Yes	4-B-7
UR-E9-(20)-1	Sequence of amino acids in wool proteins as related to quality differences	Lille, France	Yes	4-A-2
UR-E29-(20)-10	Identification of sulfur-containing compounds in wool	Leeds, England	Yes	4-A-2
UR-E29-(20)-11	Penetration of charged molecules into keratins	Leeds, England	Yes	4-A-2
UR-E29-(20)-22	Lubrication of wool knitting yarns	Nottingham, England	Yes	4-C-4
W3 1-83 (Rev.)	Flavonoids in citrus	Pasadena, Calif.	Yes	5-A-2
W3 1-88 (Rev.)	Citrus essential oils	Pasadena, Calif.	Yes	5-A-1 5-B-1
W3 1-95	Composition of dates and date products	Pasadena, Calif.	Yes	5-A-4 5-B-2
W3 1-101 (Rev.)	Fruit juice products and processes	Albany, Calif.	Yes	5-C-1
W3 1-119	Fruit flavor components	Albany, Calif.	Yes	5-A-3
WU-P-1	Plant enzymes	Albany, Calif.	Yes	5-A-5 5-A-6

[1] Project completed during reporting period.

Line Project Check List -- Reporting Period July 1, 1960 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Summary of Progress	Incl. in Area & Sub-Subheading
W3 1-67 (Rev.)	Processing quality of Northwest soft fruit and berry varieties	Prosser and Puyallup, Wash.	Yes	6-B-3 6-B-4
W3 1-101 (Rev.)	Fruit juice products and processes	Albany, Calif.	Yes	6-B-1 6-C-1 6-C-9
W3 1-102[1]	Dehydrofreezing and dehydrocanning fruits	Albany, Calif.	Yes	6-B-2
W3 1-109	Processing quality of Northwest grapes for juice	Prosser and Puyallup, Wash.	Yes	6-C-7
W3 1-110[1]	Engineering aspects of neat transfer surface fouling	Albany, Calif.	Yes	6-C-8
W3 1-112 (Rev.)	Dried fruit products and processes	Albany, Calif.	Yes	6-A-8 6-A-3 6-C-2 6-C-3 6-C-4
W3 1-113	Tree nuts products and processes	Pasadena, Calif.	Yes	6-C-6
W3 1-114[1]	Time-temperature tolerance of frozen fruits	Albany, Calif.	Yes	6-A-3
W3 1-115[1]	Softening of brined cherries	Albany, Calif.	Yes	6-C-5
W3 1-116	The chemistry of sulfur dioxide in dried fruits	Albany, Calif.	Yes	6-A-4
W3 1-117 (Rev.)	Fruit pigments	Albany, Calif.	Yes	6-A-2
W3 1-118[1]	Chemical attractant for fruit flies	Albany, Calif.	Yes	6-A-7
W3 1-119	Fruit flavor components	Albany, Calif.	Yes	6-A-1
W3 1-120 (C)	Macadamia nuts products and processes	Honolulu, Hawaii	Yes	6-C-6
W3 1-121	Heat transfer surface fouling	Albany, Calif.	Yes	6-C-8
W3 1-122	Texture of fruits and fruit products	Albany, Calif.	Yes	6-A-5 6-C-5
UR-A10-(30)-3	Microbial flora in fruits and vegetables	Rehovot, Israel	Yes	6-A-9
UR-E15-(30)-11	Canned concentrated peach and apricot purees	Parma, Italy	Yes	6-B-5
WU-P-1	Plant enzymes	Albany, Calif.	Yes	6-A-5 6-A-6
W3 4-65 (C)[1]	Potato components related to texture	Moscow, Idaho	Yes	7-A-4
W3 4-73[1]	Effects of processing on potato quality	Albany, Calif.	Yes	7-A-5 7-B-1 7-C-1
W3 4-79	Effects of processing on potato product flavor	Albany, Calif.	Yes	7-A-1 7-B-1 7-C-1
UR-E29-(30)-16	Enzymatic browning of potatoes	Cambridge, England	Yes	7-A-2
UR-E29-(30)-17	Sulfur dioxide in dehydrated vegetables	London, England	Yes	7-A-3
UR-E29-(30)-19[1]	Antioxidant activity of polyphenols	Aberdeen, Scotland	Yes	7-A-6

[1] Project completed during reporting period.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Summary of Progress	Incl. in Area & Sub-Subheading
W3 4-47 (Rev.)	Vegetable varieties	Puyallup, Wash.	Yes	8-C-2
W3 4-67	Sulfur-containing vegetable flavor components	Albany, Calif.	Yes	8-A-1 8-A-2
W3 4-69 (C)[1]	Relationship of dry bean composition to soaking and cooking time	Moscow, Idaho	Yes	8-A-6
W3 4-70 (C)[1]	Factors affecting measurement of chlorophyll	Provo, Utah	Yes	8-A-7
W3 4-71 (C)[1]	Changes in composition of bacterial spores	Urbana, Ill.	Yes	8-A-4
W3 4-72[1]	Biochemical factors of bacterial spore formation	Albany, Calif.	Yes	8-A-4
W3 4-74 (Rev.)	Composition of dry beans re processing factors and product quality	Albany and Pasadena, Calif.	Yes	8-A-5 8-A-6 8-B-2
W3 4-75	Measurement and preservation of chlorophyll in vegetables	Albany, Calif.	Yes	8-A-7 8-C-3
W3 4-77 (Rev.)	Tomato concentrate and powder	Albany, Calif.	Yes	8-B-1 8-C-1
W3 4-78 (C)[1]	Relationship of processing method to characteristics of dry bean products	Urbana, Ill.	Yes	8-A-5
W3 4-81 (C)	Dry bean characteristics	Urbana, Ill.	Yes	8-A-5
W3 4-82	Microbiology of frozen vegetables	Puyallup, Wash.	Yes	8-C-4
UR-A10-(30)-3	Microbial flora in fruits and vegetables	Rehovot, Israel	Yes	8-A-9
UR-A10-(30)-17	Sulfur dioxide in dehydrated vegetables	Cambridge, England	Yes	8-A-8
UR-E29-(30)-20	Carotenoid components of vegetables	Cambridge England	Yes	8-A-7
UR-E29-(30)-27	Relationship of composition to cooking quality of dry peas	Chipping-Campden, England	Yes	8-A-6
BF-WU-00-1	Hop oil flavor components	Albany, Calif.	Yes	8-A-3
W4 3-1	Chemical derivatives of ricinoleic acid	Albany, Calif.	Yes	9-A-3 9-B-1
W4 3-2 (Rev.)	Foamed polyurethanes from castor oil	Albany, Calif.	Yes	9-B-2
W4 3-3 (Rev.)	Pharmacology of castor bean allergens	Albany, Calif.	Yes	9-A-1 9-A-2 9-B-3
W4 3-4 (C)[1]	Preparation of castor bean allergens	Menlo Park, Calif.	Yes	9-A-1
W4 3-5 (C)	Polymerization of castor oil-derived monomers	Tucson, Ariz.	Yes	9-B-1
W4 3-6	Role of blossoms and pollen in castor allergy	Albany, Calif.	Yes	9-A-1
W4 3-7 (C)	Characterization of antigenic proteins of castor	Menlo Park, Calif.	Yes	9-A-1

[1] Project completed during reporting period.

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Work & Line Project Number	Work and Line Project Titles	Work Locations During Reporting Period	Line Proj. Incl. in Summary of Progress	Area & Sub-Subheading
W5 1-46[1]	Factors causing post-harvest composition changes in sugar beets	Albany, Calif.	Yes	10-A-1
W5 1-51[1]	Effect of molasses-forming substances on sucrose crystallization	Albany, Calif.	Yes	10-A-1 10-A-2
W5 1-52[1]	Methods for increasing the efficiency of beet juice purification	Albany, Calif.	Yes	10-A-1 10-C-1 10-C-2
W5 1-61[1]	New uses for sugar beets and constituents	Albany, Calif.	Yes	10-B-1
W5 1-72 (C)	Relation of beet composition to processing characteristics	Fort Collins, Colorado	Yes	10-A-2
W5 1-73	Biochemical studies of non-sucrose carbohydrates in sugar beets	Albany, Calif.	Yes	10-A-1
W5 1-75	Effects of non-sugar chemicals on processing	Albany, Calif.	Yes	10-A-1 10-A-2 10-C-1 10-C-2
UR-E29-(50)-33	Fatty acid esters of sucrose	London, England	Yes	10-B-1
W5 5-37	Evaluation of hydroxy-conjugated dienoic acid oils	Albany, Calif.	Yes	15-A-1 15-B-1
W6 1-48 (Rev.)	Chemistry of poultry flavor	Albany, Calif.	Yes	11-A-1 11-A-2
W6 1-49 (Rev.)	Microbiology of cold-tolerant organisms	Albany, Calif.	Yes	11-C-1
W6 1-51 (C)[1]	Factors controlling feather release	East Lansing, Mich.	Yes	11-A-4
W6 1-54	Precooked frozen foods	Albany, Calif.	Yes	11-B-2
W6 1-56	Tenderness and other textural qualities of poultry meat	Albany, Calif.	Yes	11-A-3 11-C-2
W6 1-58 (C)	Control of the neuromuscular retention and release of feathers	East Lansing, Mich.	Yes	11-A-4
QMC-0-11	Radiation preservation of poultry products	Albany, Calif.	Yes	11-B-1
W6 1-41 (Rev.)	Improvement of egg white products	Albany, Calif.	Yes	12-C-1 12-C-3
W6 1-43[1]	Flavor limitations of yolk-containing egg solids	Albany, Calif.	Yes	12-B-1
W6 1-44[1]	Deteriorative reactions of egg components	Albany, Calif.	Yes	12-A-1
W6 1-47 (C)[1]	Relationship of lipoproteins to egg product functionality	Manhattan, Kansas	Yes	12-A-2
W6 1-52 (C)[1]	Chemical characterization of yolk lipids	Austin, Minn.	Yes	12-A-3
W6 1-53	Processing characteristics of eggs	Albany, Calif.	Yes	12-A-5 12-C-2
W6 1-55	Improvement of yolk-containing egg solids	Albany, Calif.	Yes	12-C-3
W6 1-57 (C)	Oxidative changes in yolk lipids	Austin, Minn.	Yes	12-A-4
UR-E9-(60)-76	Chemistry of egg lysozyme	Paris, France	Yes	12-A-1

[1] Projected completed during reporting period.